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FURTHER STUDY OF THE USEFULNESS OF DIFFERENT TYPES OF SHORTENING WHEN INCORPORATED IN BISCUITS AT VARIOUS LEVELS AND WITH DIFFERENT BAKING TEMPERATURES[1]

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Abstract

One hundred and ninety-two 30-day-old male white rats were fed diets containing four different types of shortening (compound animal-vegetable, blended vegetable, hydrogenated vegetable, and lard) incorporated at 0, 8, 16, and 24% levels by weight and baked at 375° and 425° F. Diets were mixtures of flour, milk powder, shortening, salt, and bone meal with supplementary allowances of vitamins A, D, and B_1. The proportion of ingredients was adjusted to maintain protein at 16% by weight. The relative nutritive value of the diets was measured by growth of rats, digestibility of the diet, and the proportion of fat deposited in the livers and carcasses.

Gains decreased with increasing fat level, apparently owing to a reduction of the proportion of protein to non-protein calories from the replacement of carbohydrate by fat.

Digestibility of the fat component was unaffected by baking temperatures or level of fat in the ration. Lard was slightly more digestible than the other types which included vegetable fats. Rats fed diets baked at 425° F. made slower gains than those on the diets baked at 375° F. This was not traceable primarily to heat damage to the fat but more probably to some effect on the protein fraction.

Introduction

Owing to the influx of a variety of commercial shortenings replacing lard and butter for cooking purposes, the comparative nutritive value of these fats and of the products into which they are baked for consumption has become of renewed interest to nutritionists.

One of the factors affecting the results obtained with fat-containing diets is the amount of fat in the diet. Both Osborne and Mendel (17) and Smith and Carey (21) obtained considerably better growth in rats on fat-free diets than on combinations in which fat was included. On the other hand, Frank (5) found more rapid gains on diets containing 80% fat and 15% protein, than on "normal" rations. Osborne and Mendel (18) were unable to substantiate Frank's findings. They found that the high fat rations caused failure of appetite. This was perhaps traceable to imbalance in the diets with

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respect to essential components. Later studies (Levine and Smith (11), Hoagland and Snider (7), McAmis, Anderson, and Mendel (12)) have indicated that fat as such is not the cause of unsatisfactory nutritive values in rat diets.

The specific fat employed has not been found to be of marked importance. Most of the commonly used shortenings have shown apparent digestibility of about 95% (Holmes and Deuel (10)), which means that for practical purposes there is little difference between them in so far as energy value is concerned (Hoagland and Snider (8), Deuel, Movitt, Hallman, and Mattson (4), Deuel and Movitt (3), and Hoagland and Snider (9)).

Heating of the fats, as in baking, however, has been shown by Morris, Larsen, and Lippencott (16) to damage their feeding value for rats, while Roffo (19, 20) reports that diets containing heated fats may cause tumours in the stomach.

In a preliminary study of the nutritive value of shortenings (Crampton and Mills (2)), it was found that increasing the percentage fat of the diet at the expense of carbohydrate, on a weight basis, resulted in a sharp decline in feeding value as measured by growth of rats. The effect of a baking temperature of 425° F. for 15 min. on the complete diet was also detrimental, though this might be traceable to damage to proteins rather than to the fat fraction. These results seemed of sufficient importance that the hereinafter described test was carried out to obtain further data on this problem.

Experimental

The general plan of this test was to feed to young growing rats rations in which the four shortenings to be tested were incorporated at four different levels and the diets baked at two different temperatures.

The relative nutritive values of the final diets as fed and of the fat fractions were measured by the growth of the rats during the test period and the digestibility of the diet and diet fractions. Measurement of the fat deposition in the bodies and livers of the rats was made in the hope of obtaining further information relative to the nutritive properties of the diets.

The feeding trial involved three replicate 28-day tests, each of 64, 25- to 30-day-old, male white rats. The rats were allotted at random to individual wire-bottomed cages, where the diets and water were provided *ad libitum*. Supplements of vitamins A and D and thiamin (0.8 mgm. per week) were administered orally twice weekly.

The rations fed consisted of ingredients that could be made into edible biscuits. The percentage composition of the four different mixtures is shown in Table I.

The milk powder was increased and the flour decreased as the fat was increased in these mixtures in order to maintain a constant protein level throughout.

TABLE I

DESCRIPTION OF MIXTURES USED

Ingredient or fraction	Fat level			
	0%	8%	16%	24%
Formula				
Flour	81.5	69.5	58.5	46.5
Milk powder	16.0	20.0	23.0	27.0
Fat	0.0	8.0	16.0	24.0
Bone meal	2.0	2.0	2.0	2.0
Salt	0.5	0.5	0.5	0.5
Analysis (dry matter)				
Protein	16.0	15.8	15.4	15.6
Fat	0.0	8.6	16.6	24.6
Carbohydrate	80.2	71.6	63.7	55.6
Ash	3.8	4.0	4.3	4.2

At each fat level, four different shortenings were employed as follows:—

1. Compound animal-vegetable fat—commercial Jewel shortening, manufactured by Swift Canadian Company—48% animal fat and 52% vegetable oils. Melting point, 47.5° C.
2. All hydrogenated vegetable fat—commercial Covo S.S., manufactured by Lever Brothers Limited from peanut oil. Iodine value, 58; melting point, 42° C.
3. Blended vegetable fat—prepared in Macdonald College Chemistry Department—10% stearin and 90% soybean oil. Melting point, 47° C.
4. Pure animal fat—commercial lard.

The diets were mixed and baked into commercial biscuits at two different baking temperatures, 375° and 425° F., by Harrison Brothers Limited—the Pom Bakers, Montreal. Subsequently the biscuits were oven-dried below 100° C. and ground for feeding.

A factorial type of design was used as indicated in Appendix Table II. This design made possible a partition of the variance into that traceable to replicate tests, baking temperatures, fat levels, kinds of fat, and the interaction between them.

Individual records were kept of the gains in weight and feed consumption for the 28-day feeding periods. During the fourth week of each growth test, faeces were quantitatively collected from each rat, air-dried, and stored, pending chemical analysis, so that apparent digestibility coefficients for the diet and diet fractions could be determined.

To obtain a measure of the metabolic faecal fat for true digestibility of the shortenings the data from the rats receiving the check diets of 0% fat were

used. The average daily fat output on the fat-free diet was found to be 0.15 gm. The true digestibility of fat was calculated as:

$$100 \times \left(\frac{\text{(Dietary fat intake)} - \text{(Fat output} - \text{average metabolic fat)}}{\text{Dietary fat intake}}\right)$$

At the conclusion of the growth trial all rats were killed by stunning. The carcasses and livers were analysed for crude fatty acids by the method of Gavin and McHenry (6, 13).

Results

Because of the relation of digestibility and of carcass and liver fat deposition data to the interpretation of the growth results they will be considered first.

The mean values of the digestibility coefficients for the total diet and the energy yielding fractions are given in Table II.

TABLE II

MEAN VALUES OF PERCENTAGE DIGESTIBILITY OF DIETS AND DIET FRACTIONS

Variable	Group	% Digestibility, (to nearest whole per cent)				
		Total dry matter	Carbohydrate	Protein	Fat	
					Apparent*	True
Baking temperature, °F.	375	93	96	84	96	97
	425	93	95	83	97	98
Kinds of fat	Animal-veg.	92	95	83	95	97
	Veg. blend	93	96	84	96	98
	Hydrog. veg.	92	95	84	96	97
	Animal (lard)	94	96	83	97	99
Fat level, %	0	95	98	86		
	8	94	97	85	96	97
	16	92	95	82	96	97
	24	91	92	81	97	98
Test averages		93	96	83	96	97

* *Necessary difference for significance (P = 0.05).* *Between baking temperatures* = 0.5; *Between kinds of fat* = 0.7; *Between levels of fat* = 0.6

It is evident that baking temperature did not affect digestibility. Lard appears to have a slightly higher digestibility coefficient than the other three shortenings. This finding is in accordance with that of Hoagland and Snider (8) who reported that the digestibility of lard was superior to that of other types of shortenings. The difference is small, however, and probably of no practical consequence. For reasons not explainable in this test the apparent digestibility of the total dry matter, carbohydrate, and protein decreased and that of fat increased as the fat content of the diet increased, while the true digestibility of the fat remained constant.

In so far as the carcass and liver analyses are concerned, no pathological evidence of fatty livers was observed in any of the rats nor was there any evidence of differences in fat deposition in the carcasses except in the case of lard, which showed low values. The data are given in Appendix Table I.

In Table III the effect of the two baking temperatures on the fat deposition in rats fed the four different dietary fats is given.

TABLE III

EFFECT OF TWO BAKING TEMPERATURES ON THE CARCASS FAT DEPOSITION OF RATS FED FOUR DIFFERENT FATS

(Figures are % fatty acids in carcass)

Baking temp., °F.	Kind of fat			
	Animal-vegetable	Blended vegetable	Hydrogenated vegetable	Animal (lard)
375	8.4	7.1	8.3	4.8
425	6.0	6.4	6.0	2.9

The subnormal deposition of fat in the lard-fed rats would seem to be due to the greater adverse effect of heat on lard than on the other three shortenings. The lard diets were the only ones obviously rancid during the test. This might account for the lower fat deposition in rats fed the lard diets. Furthermore, as high temperatures are known to speed up the onset of rancidity, it is probable that this was the cause for the decreased fat deposition with the increased baking temperature.

Growth

A summary of the average initial weights of the rats, their 28-day gains in live weight and their feed consumption is presented in Appendix Table II.

In order to distinguish between differences in gains due to kinds of feed and those due to amounts of feed, the observed gains were adjusted by regression ($b = 0.299$) to average feed intake. The mean observed gains, feed intakes, and adjusted gains are given according to feeding groups in Table IV, from which the following observations can be made.

Baking Temperature

Animals on the diets baked at 375° F. showed significantly greater gains than those fed diets baked at 425° F. This, however, does not seem to be due to any effect of heat on the fat, since the greatest decrease in gains on the higher temperature occurs when there is no fat in the diet (See Table V). The inferior gains on diets baked at 425° F., compared with gains on diets baked at 375° F., therefore, would seem more likely to be due to damage to the protein at the higher temperature. This is in accord with the evidence of Morgan and King (15) who report retarded growth of rats receiving toasted bread as compared with controls fed bread crumbs.

TABLE IV

MEAN 28-DAY GAINS AND FEED INTAKE ACCORDING TO FEEDING GROUPS

(Figures to nearest gram)

Variable	Group	Observed 28-day gains	28-day feed intake	Adjusted 28-day gains	Necessary difference ($P = 0.05$)
Baking temperature, °F.	375	47	215	47	
	425	43	210	44	1.5
Kind of fat	Animal-veg.	50	226	46	
	Veg. blend	43	204	46	
	Hydrog. veg.	54	231	48	2.1
	Animal (lard)	35	190	42	
Fat level, %	0	77	297	52	
	8	41	203	44	
	16	36	186	44	2.1
	24	28	166	42	
Test average		45	213	46	

TABLE V

EFFECT ON GAINS OF DIFFERENT BAKING TEMPERATURES ON DIETS CONTAINING DIFFERENT LEVELS OF FAT

(Figures to nearest gram)

Baking temp, °F.	Fat level			
	0%	8%	16%	24%
375	56	43	44	44
425	47	45	44	40

Kinds of Fat

Diets containing the pure animal fat (lard) produced gains inferior to those produced by any one of the other three shortenings. The lower feed intake suggests a lack of palatability of the lard diets. In this connection it may be noted that the diets with lard showed an easily detected rancidity before the feeding tests were completed. Clausen, Barnes, and Burr (1) stressed the seriousness of destruction of dietary essentials and possible toxicity of rancid fats. In their studies, when lard was included in the diet with no antioxidant, rats failed to grow, and died, but if the lard were replaced by hydrogenated vegetable oils, corn oil, or butterfat, growth occurred normally.

Fat Level

Between levels of fat there are striking differences in gains as shown in Table IV. The addition of fat was associated with a sharply reduced feed consumption. We believe this to be related to the effect of fatty meals in

delaying gastric emptying time. On *ad libitum* feeding the animals eat when hungry, and probably the slower emptying time of the stomach on fatty diets decreases the frequency of hunger and thus may decrease the feed intake per day. Thus, indirectly, the addition of fat to the diet appears to have caused a drop in gains. It should be noted, however, that the efficiency of the fat-containing diets, though the same for the three levels, is below that of the fat-free ration as measured by the gains on equal feed intake.

The results of the fat level comparisons may perhaps be more clearly presented if the feed intake is expressed in terms of calories of metabolizable energy. The metabolizable energy of each diet is presented in Appendix Table III, and was determined from the composition of the diet in energy yielding fractions, and the digestibility of these components, using heats of combustion of 4.1 Cal. per gm. for carbohydrate, 9.35 for fat, and 5.65 for protein with an assumed urine calorie loss of 1.25 Cal. per gm. of digestible protein (Maynard (14)).

The metabolizable energy per 100 gm. of diet, the mean gains, the average feed intake and its equivalent in metabolizable energy, and the calories required per gram of gain, are given in Table VI.

TABLE VI

GAINS, FEED INTAKE, METABOLIZABLE ENERGY, AND CALORIES PER GRAM GAIN

Fat in diet, %	Average gain	Average feed consumed	Metabolizable* energy, Cal./100 gm.	Calories of metabolizable energy consumed	Calories per gram of gain
0	77	297	380	1129	14.7
8	41	203	417	847	20.7
16	36	186	452	841	23.4
24	28	166	487	808	28.9

* *Metabolizable energy for each diet given in Appendix Table III.*

From this table it will be seen that, as the level of fat increases, the metabolizable energy per 100 gm. of diet has increased (Column 4). But the feed intake (Column 3) has declined sufficiently to cause an actual decline in gains. The feed efficiency (Column 5), however, has also declined, so that the energy needed per unit gain (Column 6) has steadily increased with rise in fat level.

Multiple correlations indicated that about 87% of the variability in gains was traceable to variations in the intake of calories of metabolizable energy from protein, from carbohydrate, and from fat. Partial correlation indicated that the proportion of calories from protein was the principal dietary factor involved in these results. Indeed β-values* suggested that 70% of the effect of the three energy yielding diet fractions was chargeable to protein calories

* *Standard partial regression coefficient. See "Correlation and machine calculations", Wallace and Snedecor. Iowa State College of Agriculture and Mechanic Arts, Ames, Iowa.*

and only 5% to the fat level. The partial regression b^* of protein calories on gains was 0.91; of carbohydrate, 0.07; and of fat 0.03 gm.

In Fig. 1 is shown the extent to which the proportion of total calories from protein decreased with increased fat level, and its relation to 28-day gains of the rats. It will be noted that on a weight basis, the level of protein was constant at 16%.

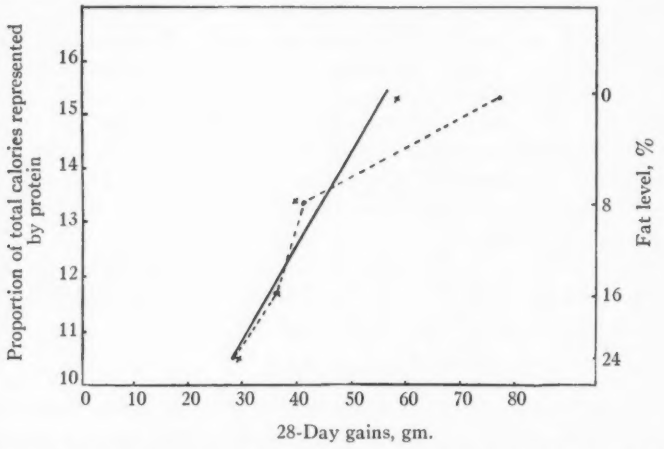

FIG. 1. *Relation of proportion of total calories represented by protein, gain, and fat level.* ------ *Observed gains.* ———— *Gains adjusted to average calories of fat diets.*

From this graph it is evident that the trend of increasing gains with increasing proportion of protein calories is practically linear between the fat containing diets, but departs significantly from this trend between the diet with lowest fat (8%) and the fat-free diet. This is traceable almost entirely to the sharply increased feed intake in the lot on the fat-free diet. Comment on the depressing effect of fat on feed consumption has already been made. If the effects of feed intake are taken into account by calculating the gain made per 832 Cal. of food (the average caloric intake of the animals on fat-containing diets) this departure from linearity disappears as shown by the broken line in Fig. 1.

Levine and Smith (11) report that as long as the proportion of protein calories to total calories was kept at about 14%, normal growth could be obtained on a high fat diet (86% of total calories).

The data of this test emphasize again the importance of balance in the ration between protein and non-protein fractions in determining the nutritive value of a diet.

Protein may be raised above minimum requirements at the expense of carbohydrate without necessarily causing any change in the energy value of

* $b_{ax} = \beta_{ax}\left(\frac{\sigma \text{ of dependent variable, } x}{\sigma \text{ of independent variable, } a}\right)$.

the diet or in resulting gains. However, if the percentage of fat is increased at the expense of protein or carbohydrate, or both, on a weight basis, the energy value of the diet is raised, but in addition the proportion of calories from protein may be decreased below that necessary for maximum nutritive value.

Summary

1. In these tests, digestibility of the fat of the diets was unaffected by baking temperature or by level of shortening in the rations. Lard was very slightly more digestible than the types that included vegetable fats. The range in coefficients was from 95.4 to 97.4% for apparent, and from 96.5 to 98.7% for true, digestibility.

2. Excepting for lard, neither type nor level of fat, nor baking temperature affected appreciably the deposition of fat in the carcasses. No evidence of fatty livers was found. Lard-fed rats showed considerably lower than average carcass fat deposition and this effect was more pronounced at the higher baking temperature. These results may reflect the effect of marked rancidity that developed with the lard.

3. Rats fed diets baked at 425° F. made considerably slower gains than those on the diets baked at 375° F. This was not traceable primarily to heat damage to the fat but was more probably the result of damage to some protein fraction.

4. Lard diets were least well eaten, with the result that they produced the lowest gains. This is probably the result of the fact that the lard became rancid.

5. Food intake was reduced by the inclusion of fat in the diets. This is believed to be due to the depressing effect of fatty meals on gastric emptying time and consequently to a reduction of the frequency of eating.

6. Observed gains and gains per unit of food intake progressively decreased with increasing fat level of the diet. This was correlated with a reduction in the proportion of calories from protein to non-protein sources, resulting from the replacement of carbohydrate yielding 4 Cal. with fat yielding about 9 Cal. per gm. Protein level on a metabolizable calorie basis may be of more nutritional significance than protein level on a weight basis if variation in fat content of the diet is involved. This in effect means that as the fat level is increased in a diet, the percentage protein, on a weight basis, must also be increased to maintain the minimum protein–non-protein calorie ratio for maximum nutritive value.

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APPENDIX TABLE I

MEAN VALUES FOR PERCENTAGE CRUDE FATTY ACIDS OF LIVERS AND CARCASSES

Variable	Group	Livers		Carcasses	
		Crude fatty acids, %	Necessary difference $P = 0.05$	Crude fatty acids, %	Necessary difference, $P = 0.05$
Baking temperature, °F.	375	2.05	0.29	7.17	0.88
	425	1.73		5.33	
Kind of fat	Animal-veg.	1.63	0.41	7.21	1.25
	Veg. blend	1.68		6.74	
	Hydrog. veg.	2.15		7.15	
	Animal	2.10		3.88	
Level of fat, %	0	1.30	0.41	6.15	1.25
	8	1.88		6.35	
	16	2.13		6.45	
	24	2.25		6.03	
Test averages		1.89		6.25	

APPENDIX TABLE II

MEAN VALUES FOR INITIAL WEIGHTS, 28-DAY GAINS, AND FEED CONSUMPTION (gm.)

Baking temp., °F.	Fat level, %	Variable recorded	Kind of fat			
			Animal-vegetable	Blended vegetable	Hydrogenated vegetable	Animal
375	0	Initial weight	51	54	51	53
		28-day gain	81	94	78	79
		28-day feed consumption	285	338	284	298
	8	Initial weight	53	57	51	49
		28-day gain	55	37	49	17
		28-day feed consumption	237	202	206	141
	16	Initial weight	54	50	47	55
		28-day gain	46	28	43	27
		28-day feed consumption	214	162	194	174
	24	Initial weight	55	48	56	46
		28-day gain	36	34	42	16
		28-day feed consumption	198	170	203	121
425	0	Initial weight	52	49	56	44
		28-day gain	80	69	73	64
		28-day feed consumption	317	273	310	253
	8	Initial weight	44	50	51	48
		28-day gain	38	46	57	28
		28-day feed consumption	185	215	241	172
	16	Initial weight	58	45	51	52
		28-day gain	41	19	51	32
		28-day feed consumption	217	133	214	182
	24	Initial weight	51	51	47	58
		28-day gain	27	16	36	17
		28-day feed consumption	161	140	181	155

APPENDIX TABLE III

METABOLIZABLE ENERGY OF DIETS

Baking temp., °F.	Fat level, %	Type of fat			
		Compound animal-vegetable	Blended vegetable	Hydrogenated vegetable	Animal
375	0	380*	382	381	381
	8	417	411	418	428
	16	451	445	451	466
	24	481	483	483	506
425	0	380	379	379	379
	8	415	413	416	421
	16	446	448	449	460
	24	483	482	482	494

** Cal./100 gm. diet.*

DRIED WHOLE EGG POWDER

XIX. ACCELERATED STORAGE TESTS TO ASSESS THE EFFECT OF HEAT TREATMENT, MOISTURE CONTENT, AND MATERIALS ON THE QUALITY OF DRIED SUGAR–EGG MIXTURES[1]

By R. L. Hay[2] and Jesse A. Pearce[2]

Abstract

Deterioration in quality was assessed by fluorescence, potassium chloride, pH, and foaming volume measurements.

Dried egg powder (moisture content, 2.8%), containing 33% sugar, and control samples of plain egg powder (moisture content, 3.9%) were stored at temperatures of 80°, 100°, 120°, and 140° F. for seven days. At 140° F. the addition of sugar inhibited the initial, but not the secondary, fluorescence development observed in the plain egg powder and retarded deterioration as assessed by other measurements. At temperatures of 120° F. and lower, the presence of sugar had a marked effect in retarding decrease in quality in egg powder as assessed by all quality tests used. Interpretation of the results in terms of commercial drying practices indicated that cooling shortly after drying was less important for sugar–egg powder than it was for plain egg powder.

Dried egg powder containing 33% sugar was adjusted to moisture levels of 1.4, 2.8, and 3.2% and stored at 80° and 120° F. for seven days. The rate of deterioration in quality of sugar–egg powder increased markedly with both moisture content and temperature. Egg powder containing 1.4% moisture maintained higher quality at both temperatures for a longer period than powders at either 2.8 or 3.2% moisture levels. It is recommended that sugar–egg powder be dried to the lowest moisture content compatible with the production of good quality powder, certainly to a moisture content of less than 2.8%, and preferably to 1.4%.

Loss in quality was less for sugar–egg powders (moisture content, approximately, 2.3%) prepared with granulated sugar than for those prepared with sucrose syrup, when stored at 80°, 100°, 120°, and 140° F. for seven days. In addition, powder made from fresh shell eggs was more desirable than powder prepared from frozen melange. It is recommended that sugar–egg powder be prepared from a mixture of sugar in granulated form and melange from fresh shell eggs.

Introduction

Eggs in powdered form have become a well known commodity during the war years. The production of egg powder in Canada during pre-war years was almost negligible but it is probable that the demand for this product will be increased during the post-war period. However, the extent to which this commodity competes successfully with other egg products during normal times will depend largely on how well its quality can be maintained, not only during production but also during subsequent handling and storage.

In a recent communication it was observed that the addition of sucrose to egg powder, prior to drying, was effective in delaying fluorescence development at temperatures of 118° F. and lower (9). Present indications are that this sugar–egg powder will find a ready peacetime market for baking and other trade purposes.

[1] *Manuscript received September 29, 1945.*

Contribution from the Division of Applied Biology, National Research Laboratories, Ottawa. Issued as paper No. 156 of the Canadian Committee on Food Preservation and as N.R.C. No. 1382.

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Previous investigations have shown that temperature and moisture content were major factors in the preservation of dried foods. Earlier studies showed that rapid cooling of plain egg powder to a temperature of 80° F. or less within three hours of leaving the drier was important if high quality were to be maintained (14, 15, 16). Lowering the moisture content prolonged the storage life of this powder (7, 15) but even at the low level of 1.4% moisture, dried egg deteriorated in a relatively short time when stored at temperatures of 99° and 118° F. (13). Moisture levels of 2% and less were believed desirable, but Canadian commercial drying operations did not permit the attainment of these lower levels.

Some sugar–egg powder is being produced in Canada at the present time. Current drying practice favours the production of sugar–egg from melange to which sucrose syrup rather than granulated sucrose has been added, although no comparison of the relative qualities of the final products has been made. Furthermore, no attempt has yet been made to compare the quality of sugar–egg powder prepared from shell eggs with that prepared from frozen melange.

Since sugar in egg had an inhibitory effect on fluorescence development, it was of interest to determine whether sugar–egg was as susceptible to heat deterioration as plain egg powder, to compare the effect of various moisture contents on the quality of sugar–egg powders, and to make some assessment of the function of sugar in this product. This report also describes the effect of heat treatment on sugar-egg powders produced from the following: (*a*) shell eggs and sucrose syrup, (*b*) shell eggs and granulated sucrose, (*c*) frozen melange and sucrose syrup, and (*d*) frozen melange and granulated sucrose.

Comparisons of plain and sugar–egg powder in this study were made on the assumption that the moisture was almost entirely contained in the egg fraction of the sugar–egg. Reference to equilibrium relative humidity data for egg powder showed that, at room temperature and about 3% moisture, the relative humidity over a sample of plain egg powder would be about 15% (5). Reference to similar data (4) for sugar at 25° C. indicated that commercial sucrose (containing 0.05 to 0.2% invert sugar) would not begin to take up moisture until a relative humidity of about 70% was reached. Even if inversion occurred during the drying process, it is unlikely that moisture would be elsewhere than in the egg powder, since sucrose containing 10% invert sugar does not begin to take up moisture until a relative humidity of about 25% is reached.

Materials and Methods

The powder containing 33% sugar (dry matter basis) and the plain egg powder used in the heat treatment study were obtained from the same commercial Canadian source. These samples were tempered in the laboratory to a moisture content of approximately 4%, calculated on the basis of egg solids. The actual moisture content of the sugar–egg was 2.8%; the plain egg, 3.9%. The sugar–egg powder used in the moisture study was obtained from the same Canadian producer, and was tempered to moisture levels of 1.4, 2.8,

and 3.2%. Calculated on the basis of egg solids, the moisture contents were 2.1, 4.0, and 4.6% respectively. After the moisture adjustment had been completed samples of these powders were packed in tin-plate (air as headspace gas), stored at temperatures of 80°, 100°, 120°, and 140° F. and removed for analysis after one, two, five, six, and seven days.

The powders prepared from various mixtures (sugar content, 33%, dry weight basis) were obtained from another Canadian producer and adjusted in the laboratory to approximately the same moisture level (actual moisture content between 2.2 and 2.4%). Samples of these powders were also packed in tin-plate (air as headspace gas), stored at 80°, 100°, 120°, and 140° F., and removed for analysis after one, two, three, four, five, six, and seven days.

All powders were prepared on a cone type spray-drier. The choice of producers was a matter of convenience only. The drying temperatures were those believed most desirable for the respective pieces of equipment.

The quality of the powders was evaluated by measurement of fluorescence (8), potassium chloride value (12), pH (12), and foaming volume; the last was believed to provide a method of assessing the baking quality. The procedure for measuring the foaming volume of the plain egg samples was the same as that reported in an earlier study (11). For the sugar–egg samples, the foaming volume was assessed as follows: 40.5 gm. of the powder was mixed thoroughly with 75 ml. of distilled water. Best results were obtained by adding a small amount of the water to the powder, whipping the mixture manually into a thick homogeneous paste, and then pouring in the remainder of the water. The mixture was beaten at the highest speed in a "Mixmaster" for 10 min., and the volume of the foam measured in a graduate. Although the two methods used were not comparable, it was felt that relative differences occurring during storage would be apparent.

Results

The Effect of Heat Treatment

The relative effects of temperature and storage time on the quality of both types of egg powders were evaluated by means of analyses of variance. The significant results are shown graphically in Figs. 1*a* and 1*b*. Fluorescence values increased and potassium chloride values, pH values, and foaming volume decreased with both storage temperature and time.

At least two separate reactions may occur during the development of fluorescing materials in plain egg powder, since the previous curves have shown a definite break or irregularity during the formation of fluorescing substances (14). This has suggested the possible presence of an initial and a secondary reaction. In the present study, curves for the fluorescence development in materials containing no sugar showed evidence of two reactions, but the presence of sugar appeared to retard the initial reaction.

At temperatures of 120° F. and lower, sugar had a marked effect in reducing the rate at which fluorescence developed. Sugar–egg at 140° F. attained the

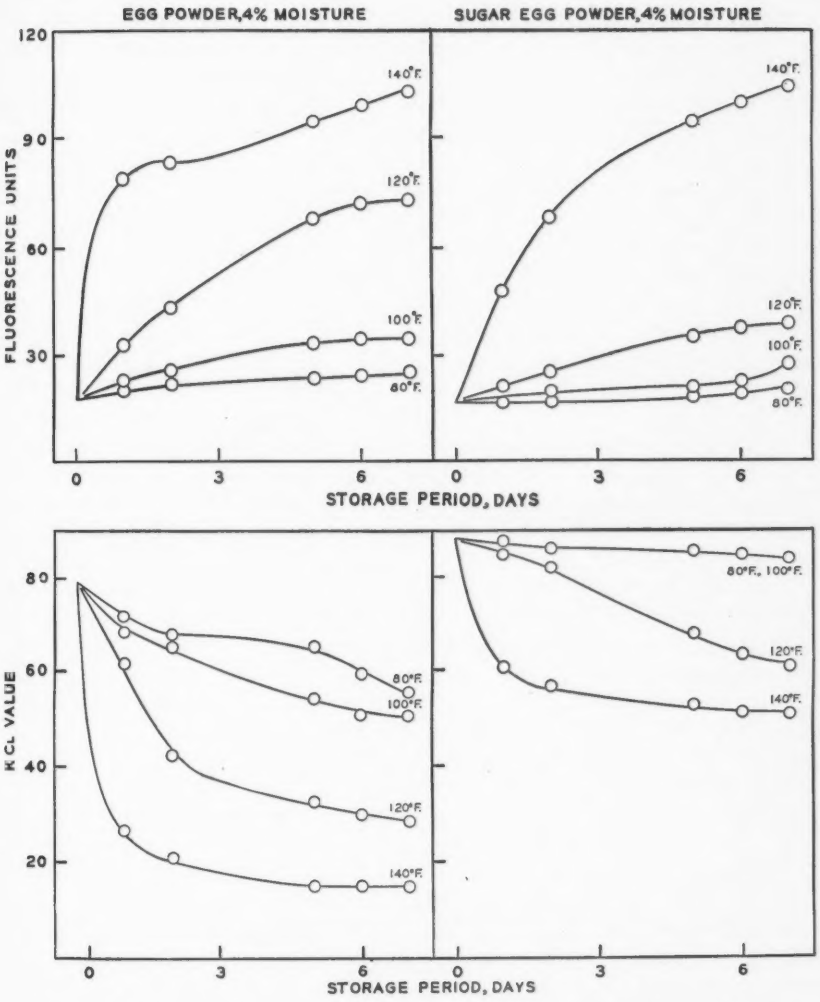

FIG. 1*a*. *Effect of heat treatment on fluorescence values and potassium chloride values of plain and sugar–egg powders with a moisture content of 4%, calculated on the basis of egg solids present. (Actual moisture content of sugar–egg powder, 2.8%.)*

same maximum value as plain egg, but the curve indicated retardation, elimination, or alteration of the first stage of the fluorescence reaction (Fig. 1*a*). At 140° F., the presence of sugar reduced the development of fluorescence by about one-half after storage for one day.

Earlier work had shown that sugar reduced fluorescence development during the first two weeks' storage at 118° F. and during eight weeks' storage

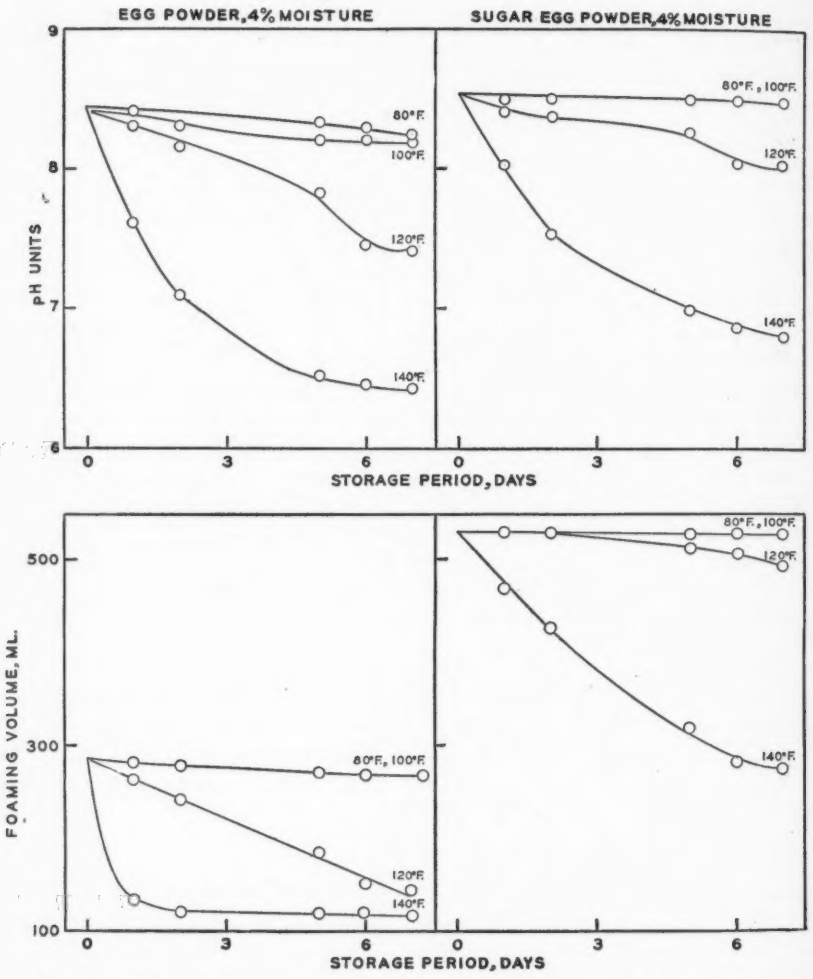

FIG. 1*b*. *Effect of heat treatment on pH and foaming volume of plain and sugar–egg powders with a moisture content of 4%, calculated on the basis of egg solids present. (Actual moisture content of sugar–egg powder, 2.8%).*

at lower temperatures (9). The present study showed that sugar effectively retarded fluorescence development during seven days' storage at 80°, 100°, and 120° F.

The maximum fluorescence value allowable for Canadian Grade *A* plain egg powder has been established at 24, and that for Grade *B* at 57 (3). Prime quality sugar–egg powder stored at 80° F. remained at a lower fluorescence level than that of plain egg during the entire period of seven days (Fig. 1*a*).

At 100° F., high quality was maintained in the sugar–egg five days longer than in the non-sugar powder. Best quality sugar–egg powder stored at 120° F. decreased to Grade *B* quality in 48 hr., while only about eight hours were required for a similar deterioration in plain egg powders. At 140° F., sugar appeared to have an inhibitory effect sufficient to delay deterioration for several hours.

Fluorescence development as depicted in Fig. 1*a* revealed that sugar added to egg powder before drying provided a beneficial effect on quality. Since sugar–egg powder was less susceptible to heat treatment it appeared that cooling to 80° F. shortly after drying would not be as important for sugar–egg as it is for plain egg.

The solubility of plain egg powders in a 10% potassium chloride solution decreased when either temperature or storage time was increased, and this was believed due to thermal decomposition of a fat–protein complex and denaturation of the egg protein (14).

In this study (Fig. 1*a*), the solubility of plain egg decreased during storage at all temperatures, the most marked change occurring in the first two days at 140° and 120° F. Subsequent changes at these temperatures were relatively slow; this indicated that the denaturation processes may have been approaching completion. The presence of sugar in egg powder appeared to provide the most protection at 80° and 100° F., and a comparison of the curves for sugar–egg and plain egg powders at 120° F. also showed evidence of some preservative action.

Correcting the potassium chloride values of sugar–egg powder for the presence of added sugar indicated that about 80% of the egg solids were soluble, which is about the same quantity usually soluble in fresh plain egg powder. After two days at 140° F., plain egg powder was only 20% soluble, but the egg fraction of sugar–egg powder was still about 40% soluble. This indicated that the presence of sugar in some way retarded or impeded thermal decomposition of the fat–protein complex or protein denaturation.

It has been reported that a decrease in pH accompanied quality deterioration in egg powders (12, 14). Further evidence of this is apparent in Fig. 1*b*. The pH of the plain egg powder decreased at all temperatures, with the greatest reduction occurring during the first five days at 140° F. The stabilizing effect of sugar as indicated by an almost constant pH was most evident at temperatures of 80° and 100° F. Sugar had a slightly beneficial effect at 120° F., but less at 140° F. These observations agreed with, and supported, the results obtained from the more sensitive fluorescence and potassium chloride tests.

For the plain egg powder the decrease in foaming volume at both 80° and 100° F. was relatively small, and of approximately the same order of magnitude. At 120° F. the decrease in this attribute was greater than at the lower temperatures, but the greatest change occurred in the plain egg during the first 24 hr. at 140° F. The subsequent changes at the high temperature were extremely slow; this indicated that the components responsible for the foaming property had been altered rapidly.

Although the techniques for determining foaming volume differed for plain and sugar–egg, and a marked increase in foaming volume is known to result from the addition of sugar to egg powder prior to drying, it was felt that the relative changes noted here were comparable (1). At 80° and 100° F. the foaming volumes of sugar–egg remained approximately equal to the initial value during the entire storage period. At 120° F. the foaming volume of sugar–egg powder decreased only 46 ml. after seven days in storage. Under the same conditions, the foaming volume of the plain egg powder decreased 145 ml. Thus, sugar had marked effect in maintaining high foaming volumes at 120° F. At 140° F. total loss of the components responsible for the foaming quality appeared to have been postponed several days owing to the presence of sugar.

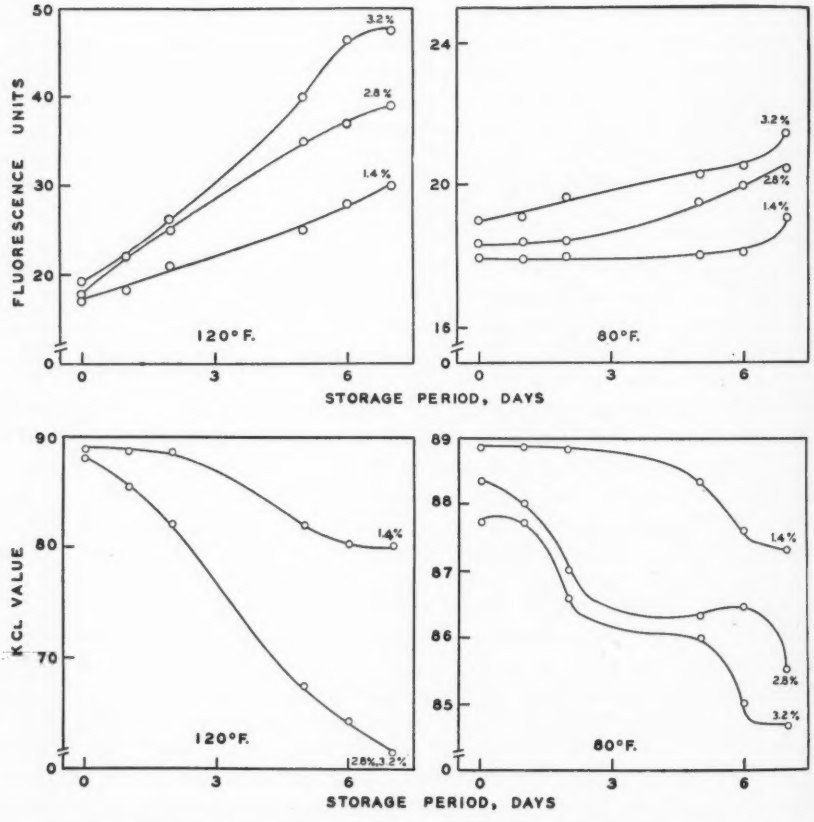

FIG. 2a. *Effect of heat treatment on fluorescence values and potassium chloride values of sugar–egg powder having actual moisture contents of 1.4, 2.8, and 3.2%.*

The Effect of Moisture Content

The effect of moisture content on the quality of sugar–egg powders was also evaluated by means of analyses of variance, and the significant results are shown graphically in Figs. 2*a* and 2*b*. The general effects of temperature and storage time noted in the heat treatment study were again observed in this study. Powder containing 1.4% moisture was better initially, except for foaming volume, and remained superior to the other powders during storage. The 3.2% powder produced the highest initial foaming volume but after the sixth day in storage deteriorated to a level below that of the lower moisture powders.

The rate at which the fluorescence value increased was roughly proportional to the moisture content. At 80° F., egg powder with a 3.2% moisture

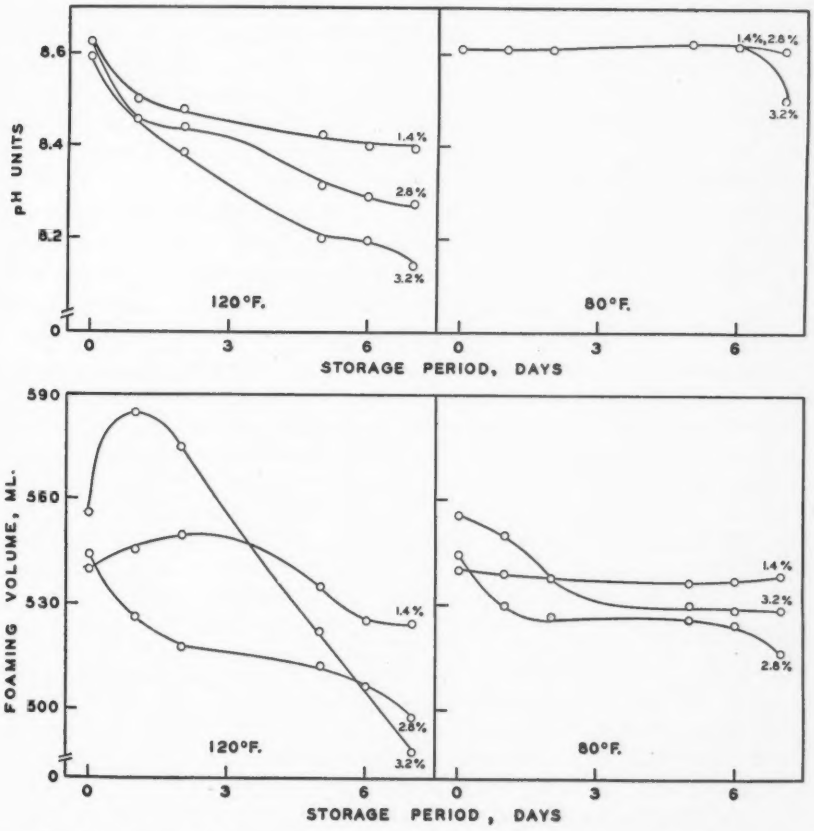

FIG. 2*b*. *Effect of heat treatment on pH and foaming volume of sugar–egg powder having actual moisture contents of 1.4, 2.8, and 3.2%.*

content remained at a higher fluorescence level than either the 1.4 or 2.8% powders throughout the storage period of one week. At 120° F., powder containing 1.4% moisture had an initial fluorescence value of 17 and this increased linearly to a final value of 30 after the seventh day of storage. The 2.8 and 3.2% powders deteriorated at approximately the same rate during the first few days but attained final fluorescence values of 39 and 48, respectively. The 2.8 and 3.2% powders had attained a fluorescence value of 24, the maximum value allowable for Canadian Grade *A* egg powder (3), after two days of storage at 120° F., while five days were required for a similar deterioration in the 1.4% powder; this indicated that drying sugar–egg to a low moisture content might extend the life of the product about two and one-half times.

Egg powder containing 1.4% moisture and stored at 80° F. for seven days had a higher solubility than either the 2.8 or 3.2% powders. The solubility of the egg powders at all three moisture levels decreased more rapidly during storage at 120° F. than during storage at 80° F., with the most marked decreases occurring in powders with moisture contents of 2.8 and 3.2%. A comparison of the curves (Fig. 2*a*) for powders stored at 120° F. showed that loss in solubility in the 1.4% powder was approximately one-third that in the 2.8 and 3.2% powders after storage for one week.

Sugar–egg powders containing 1.4% moisture maintained higher pH values for a longer period than those powders at either 2.8 or 3.2% moisture levels (Fig. 2*b*). At 120° F., the decrease in pH was most rapid for the powder with 3.2% moisture. The powder with 1.4% moisture maintained the highest pH level during the entire storage period.

These results of foaming volume measurements on the stored powders were slightly different from those shown by the previous measurements. During the first few days 3.2% moisture appeared to be most desirable but after seven days the foaming volumes of this powder had decreased rapidly to a level below that of the 1.4 and 2.8% powders. The curves indicated that this initial beneficial effect was of short duration and that after an extended storage period the powder containing the lowest moisture would be most desirable. Extended studies on the change in foaming volumes of sugar–egg powders are currently under investigation in these laboratories.

Effect of Materials Used

The effects of temperature, storage time, method of adding sugar, and prior condition of melange, on quality of the sugar–egg powder were also evaluated by means of analyses of variance. Although initially the powders did not differ in quality, every test used on the stored samples showed significant differences to result from the different materials used (Tables I and II). Differential behaviour of significance is shown in Figs. 3 and 4. Both the tables and the figures contain mean values of each variable, as calculated over all others for the whole experiment.

TABLE I

EFFECT OF HEAT TREATMENT ON SUGAR–EGG POWDERS PREPARED FROM SHELL EGGS AND FROM FROZEN MELANGE

Testing method	Mean value for powder prepared from			
	Shell eggs		Frozen melange	
	Initially	After storage*	Initially	After storage*
Fluorescence value	20.0	30.2	22.2	27.4
Potassium chloride value	86.4	79.3	87.3	77.8
Foaming volume	632	611	602	568
pH value	8.64	8.42	8.57	8.36

* *Averaged over all storage times and temperatures.*

TABLE II

EFFECT OF HEAT TREATMENT ON SUGAR–EGG POWDERS PREPARED WITH SUGAR AND WITH SYRUP

Testing method	Mean value of powder prepared with			
	Granulated sugar		Syrup	
	Initially	After storage*	Initially	After storage*
Fluorescence value	20.5	28.2	21.7	29.6
Potassium chloride value	87.3	79.2	86.0	77.9
Foaming volume	638	623	600	556
pH value	8.64	8.44	8.56	8.34

* *Averaged over all storage times and temperatures.*

The rate of deterioration in all powders increased with both temperature and storage time (Fig. 3), and the trends observed were similar to those noted in the earlier study (Figs 1*a* and 1*b*). Comparison of the curves in Fig. 3 with those in Figs. 1*a* and 1*b* shows the beneficial effect of a reduction of 0.5% in moisture content.

The initial measurements showed small differences between the various samples of the dried product, and all but the fluorescence measurements on the stored powders supported the evidence that shell egg produced a better dried material than frozen melange (Table I). However, another investigation in these laboratories has shown that freezing reduces the fluorescence of liquid egg (10) and also reduces the intensity of the light given off by fluorescing materials isolated from egg powder (2). Therefore, the low fluorescence values for stored powders prepared from frozen material do not necessarily indicate high quality. The superiority of shell egg over frozen egg was shown most markedly by potassium chloride and foaming volume measure-

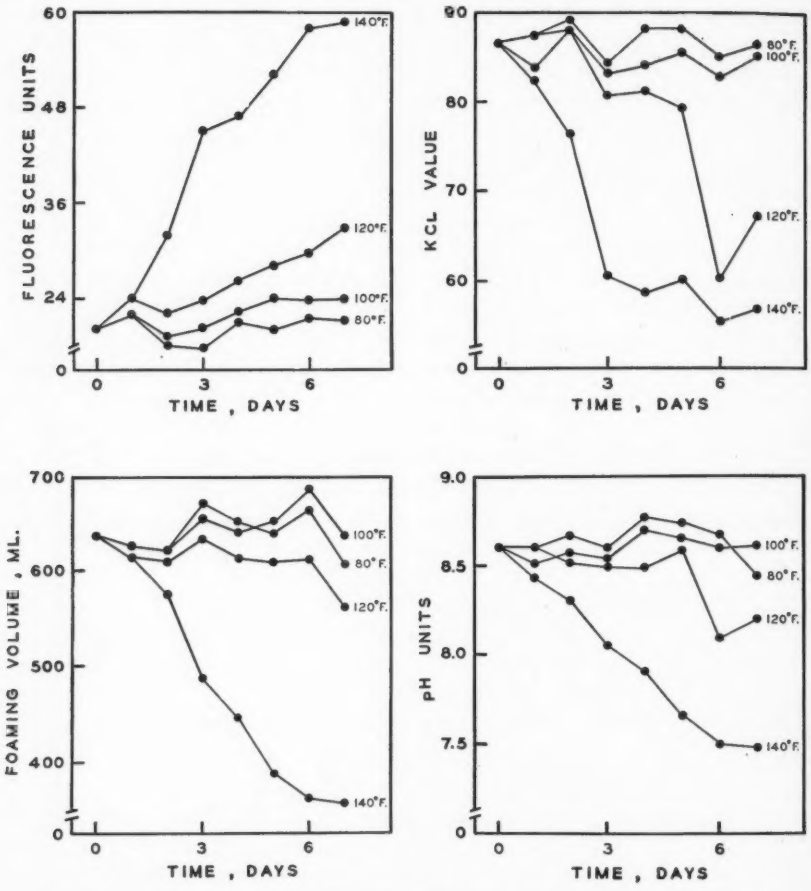

FIG. 3. *Average effect of heat treatment on fluorescence values, potassium chloride values, pH, and foaming volume of sugar–egg powder prepared from shell and frozen eggs using sucrose crystals and sucrose syrup. Actual moisture content of these samples was about 2.3%. Comparison with Fig. 1a indicates the improvement resulting from 0.5% reduction in moisture content.*

ments. Freezing the melange apparently made some of the soluble constituents, possibly those responsible for the aerating properties in egg, less stable. This is receiving further consideration in these laboratories.

Sugar–egg powder prepared by adding sugar appeared slightly better initially than the product prepared from syrup, and when the powders were stored these differences became significant as assessed by all measurements (Table II). The initial 38 ml. difference in foaming volume values was considered most important, especially as sugar–egg is used only for baking. In addition, this difference was accentuated by the storage treatment.

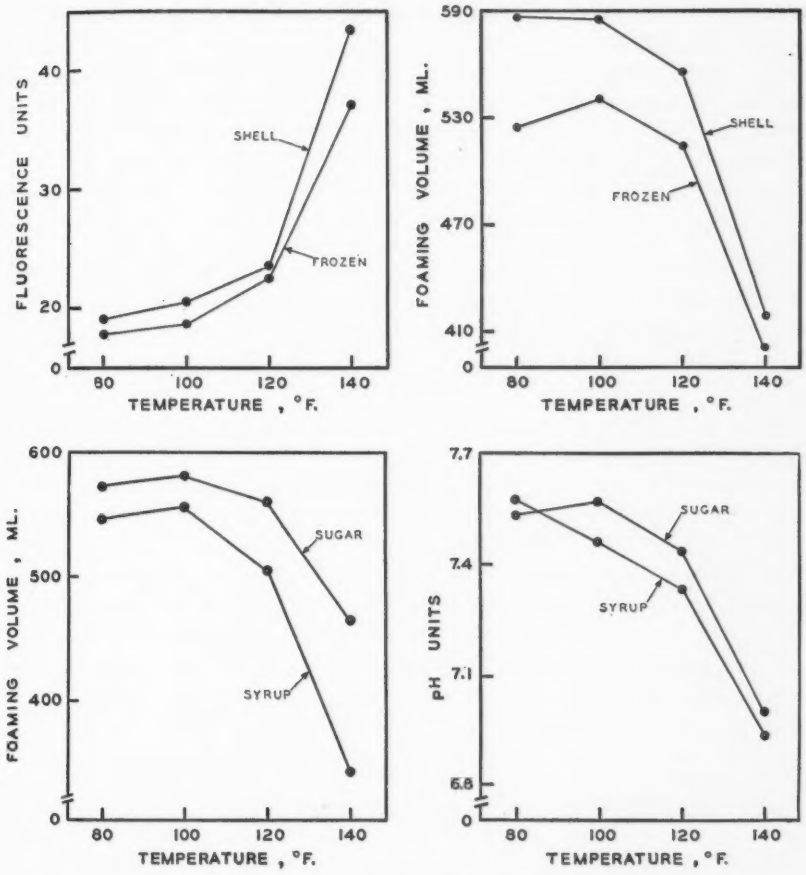

FIG. 4. *Average effect of temperature on some of the quality measurements applied to sugar–egg powder prepared from shell and frozen eggs, using sucrose crystals and sucrose syrup. Actual moisture content of these samples was about 2.3%.*

The effect of temperature on the fluorescence and foaming volume measurements of sugar–egg powders made from shell egg and from frozen melange, is presented graphically in Fig. 4. Powder produced from frozen melange had lower fluorescence values at all storage temperatures studied. At 80°, 100°, and 120° F. increases in fluorescence were slight and approximately parallel, while at 140° F. both showed very rapid increases in fluorescence development. The divergence of the curves indicated that powder from frozen melange developed fluorescing substances more slowly as the storage temperature was increased from 120° to 140° F. The foaming volume curves showed the pronounced superiority of shell eggs over frozen melange as a

component of sugar–egg powder when subjected to storage temperatures of 80°, 100°, and 120° F.

Sugar–egg powder prepared with syrup and stored at elevated temperatures was more susceptible to deterioration than the product made with granulated sugar (Fig. 4). Although the foaming volume of the latter product, after storage at 80° and 100° F. was better by only about 35 ml., this difference progressively increased to approximately 125 ml. at 140° F. Since a decrease in pH also accompanies quality deterioration it is evident from the pH curves that egg powder prepared with syrup deteriorated more rapidly than that prepared with sugar, when the powders were stored at 100° and 120° F. At 80° and 140° F. the difference between mean pH values, although favouring the powder prepared with sugar, was very small.

Discussion

The results of the heat treatment study show that the addition of sugar to egg prior to drying helps to maintain those qualities desirable for baking. Cooling after drying was less important for sugar–egg powder than it was for plain egg. Nevertheless, it is believed desirable to maintain the cooling practices in current use by industry.

The results of the moisture study indicated that the water content of sugar–egg powder should be below 2.8% and preferably about 1.4% if quality comparable to that of fresh egg powder is to be maintained during storage.

For comparison of the effect of moisture content in sugar–egg powder there was no need to correct fluorescence and potassium chloride values for the presence of added sugar, since all measures were relative. However, to compare plain and sugar–egg powders some adjustment was necessary. This has been done for the potassium chloride values shown in Fig. 5. Since the increase in fluorescence due to the caramelization of the sugar was difficult to evaluate, no correction was made for the fluorescence values of sugar–egg powder. However, it was believed that even if this correction were made the fluorescence curves in this figure would be only slightly altered and the conclusions would be much the same.

Since the sugar in the dried product can be assumed to have a negligible moisture content, all the moisture in sugar–egg powder is probably in the egg fraction. It is possible that the moisture may be equally distributed throughout both sugar and egg fractions, and the beneficial effects attributable to sugar may be due to the ability to dry to a low moisture content, thereby reducing the moisture in the egg fraction. The solid circles and squares shown in Fig. 5 represent an interpolated value for plain egg powder at 3.5% moisture and 120° F. from data previously reported (14) and an actual value for plain egg powder at 3.9% moisture and 120° F. from the heat treatment study, determined after two days' storage. The fluorescence increment and potassium chloride decrement of sugar–egg powders indicated that this product when stored at 120° F. changed by about the same amount as plain egg

powder stored at 110° F. for the same time and certainly much less than can be expected for plain egg powder stored at 120° F. This comparison indicates that sugar exerts a pronounced beneficial effect separate from any suggestion of low moisture content in the egg fraction attributable to distribution of the moisture between the egg solids and the sugar solids.

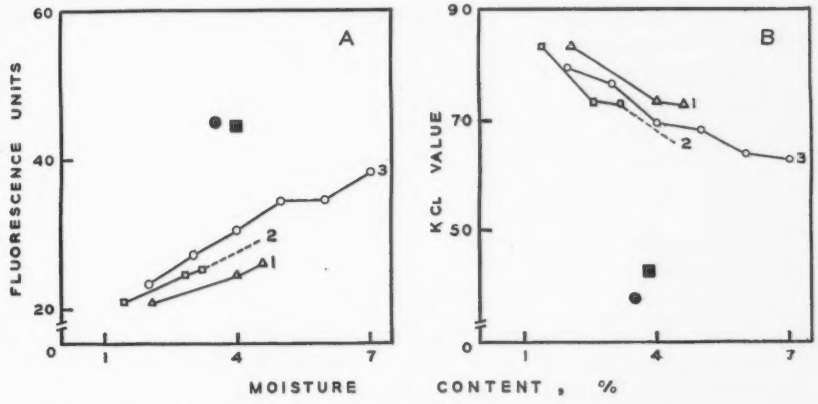

FIG. 5. *Effect of moisture content and added sugar on the fluorescence and potassium chloride values of dried egg powder after two days' storage. Curve 1—sugar–egg stored at 120° F.; moisture content calculated on basis of egg solids. Curve 2—sugar–egg (actual moisture content) stored at 120° F. Curve 3—plain egg powder (actual moisture content) stored at 110° F. (15).* ■ *Plain egg powder, 3.9% moisture, from heat treatment study, storage temperature 120° F.* ● *Plain egg powder, 3.5% moisture, value interpolated for 120° F. from data previously reported (14).*

Some physical or chemical combination may occur between sugar and the components of the egg, and provide protection to the product not only during the drying process but during subsequent handling. Although the nature of this combination is at present in doubt, the practical aspects are of significance, and are receiving further attention in these laboratories.

The results of the study on powder prepared in different ways showed that the storage life of sugar–egg powder was improved by adding granulated sugar instead of syrup to the liquid egg prior to drying. This may be explained on the basis of drying operations. The mixture made with syrup had a higher moisture content than the mixture prepared with solid sugar. Therefore, to obtain the same production rate in terms of solids requires a higher drying temperature (4, 12, 17), and corresponding deterioration in the product would be expected. To obtain drying at the same inlet and outlet temperatures necessitates reduction in melange-input. If it is assumed that the liquid particles from the egg–syrup mixture are identical in size with the liquid particles from egg–sugar mixture, the dried particles would be smaller, settle more slowly and, as a result, may be exposed to a longer period of heating in the drier, thus causing reduction in quality of the product.

This study also showed that melange from fresh shell eggs produces a sugar–egg powder superior to that from frozen melange. Egg melange is a colloidal complex containing in solution proteins, fats, a trace of sugar, lecithin, and about 1% of salts. It is known that colloids when frozen do not normally recover their original state on thawing, a phenomenon that is probably due to precipitation or coagulation of the proteins during freezing. It has been observed that egg yolk, frozen, stored below −6° C. for a reasonable time and then thawed, lost its fluidity and passed into a viscous condition with a reduced volume (6). A similar treatment caused the white to separate into liquid and viscous parts with the former increasing at the expense of the latter by an amount depending upon the temperature and storage time. The rate of freezing and thawing of the egg has also been found to affect the composition of the resulting melange (6). These changes may be responsible, in part at least, for the differences in behaviour of powders prepared from shell egg and from frozen melange.

Acknowledgments

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LIQUID AND FROZEN EGG

II. METHODS OF DETERMINING SOLIDS CONTENT OF LIQUID AND FROZEN EGG[1]

By C. G. Lavers[2] and Jesse A. Pearce[3]

Abstract

Solids content of liquid egg prepared from shell eggs having different histories was measured by the official A.O.A.C. vacuum oven method and compared with measurements of specific gravity and refractive index. Specific gravity measurements were the least satisfactory, but may provide a rough check on solids content. Refractive index measurements following treatment with ammonium hydroxide were a more satisfactory measure of solids content than the same measurement on liquid egg after treatment with trypsin solution.

Relations between the solids content of defrosted frozen egg and unfrozen liquid egg and refractive index, as determined with a Zeiss sugar refractometer and with a hand sugar refractometer, were calculated for the method involving the addition of ammonium hydroxide. The relation, solids–refractive-index, for unfrozen liquid egg differed from the relation for frozen egg. However, the method provided a rapid, convenient, and accurate means for determining solids content.

Introduction

In recent years dried egg production has increased markedly. Since drying capacity is limited, all the eggs produced during the peak laying season cannot be dried or used immediately. Much of this egg is frozen and held in storage for subsequent drying, or for use as thawed liquid egg by bakers and others. One problem of importance in drying and freezing liquid egg is that of a rapid test for the solids content. The use of refractive indices for this purpose has been described (4). The present paper compares two of these refractometric methods (2, 4) and a hydrometric method with the official A.O.A.C. vacuum oven method (1, p. 308).

Materials and Methods

Preliminary experiments indicated that liquids prepared from eggs having different histories differed in their initial solids content, and in their behaviour on dilution. Therefore, dilutions of liquid prepared from eggs with a variety of histories were used in this study. To compare the various methods, the following eggs were used: currently available Grade *A* large summer eggs; Grade *A* large summer eggs obtained within one day of laying; Grade *A* large spring eggs, stored at 0° C. (32° F.) for three months (all grades refer to condition of eggs before storage); Grade *A* large spring eggs held in commercial storage for three months; Grade *A* large spring eggs stored at 0° C. for three months followed by two weeks at 21.1° C. (70° F.); currently available

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Grade *A* pullet summer eggs; and Grade *C* summer eggs. To obtain a more representative sample, eggs of the following grades were added to the above in determining the final relation between refractive index and solids content; Grade *A* large early spring eggs; Grade *A* pullet spring eggs; Grade *C* spring eggs. In addition, frozen egg of five different grades, ranging from top quality to mouldy, was used in the investigation.

All samples of both liquid and defrosted frozen egg were prepared for analysis by mixing with a "Mixmaster" at low speed. The official A.O.A.C. vacuum oven method for determining solids content (1, p. 308) was used as a standard against which the various methods were compared.

Specific gravities were determined using a hydrometer having a range of 1.000 to 1.070 (60°/60° F.). In making the determination, it was found necessary first to remove the foam from the surface of the liquid egg, allow the hydrometer to sink gently in the liquid to a steady position, then push it down one or two scale divisions and let it rise to an equilibrium position. Through a series of 35 determinations at temperatures ranging from 10° to 35° C. (50° to 95° F.) the temperature gradient was observed to be −0.00032 specific gravity units per °C. rise in temperature (−0.00018 units per °F.). Using this figure, all determinations were corrected to give specific gravity at 15.6° C. (60° F.).

The two refractometric methods used have been described (2, 4). One of these depended on an enzymatic (trypsin) digestion (4), the other on the use of an electrolyte, 28 to 29% reagent grade ammonium hydroxide (2). The former method involved the use of a trypsin solution (trypsin, 500 gm.; water, 770 ml.; and 0.25 *N* sodium hydroxide, 800 ml.), having a refractive index of 1.377 at 30° C. To 10 gm. of whole egg, 1.8 ml. of this solution was added, followed by thorough mixing. Several drops of the mixture were placed on the prisms of the refractometer and after 30 sec. the refractive index was read. The latter method required the addition of 10 drops of ammonium hydroxide to 20 ml. of the egg sample, mixing, and determination of the refractive index as before. All refractive indices were determined at 30° C. ± 0.5° C.

For the major portion of this work a Zeiss sugar refractometer was used. In addition, the use of a hand refractometer was considered as a more convenient method for plant purposes. This instrument was a Bausch and Lomb hand sugar refractometer, reading from 0 to 60% sugar.

Results

Comparison of Methods

Refractometric and hydrometric measurements showed a high correlation with solids content as determined by the official A.O.A.C. vacuum oven method. Equations expressing the relation between these rapid determinations and per cent solids in liquid egg, and estimations of the error that would be involved in using them to predict solids content, are given in Table I. While the equations relating solids content in any one type of egg to the other measurements differed somewhat in slope (Figs. 1, 2, and 3), these differences

TABLE I

EQUATIONS, WITH ERRORS OF PREDICTION, RELATING PER CENT SOLIDS IN LIQUID EGG (y) TO VALUES BY RAPID METHODS (x)

Method	Equation	Error of prediction, % solids
Specific gravity (hydrometric)	$y = 793.02\,x - 794.77$	$\pm$ 1.3
Refractive index (enzymatic)	$y = 678.04\,x - 907.94$	$\pm$ 0.66
Refractive index (electrolytic)	$y = 574.75\,x - 766.02$	$\pm$ 0.42

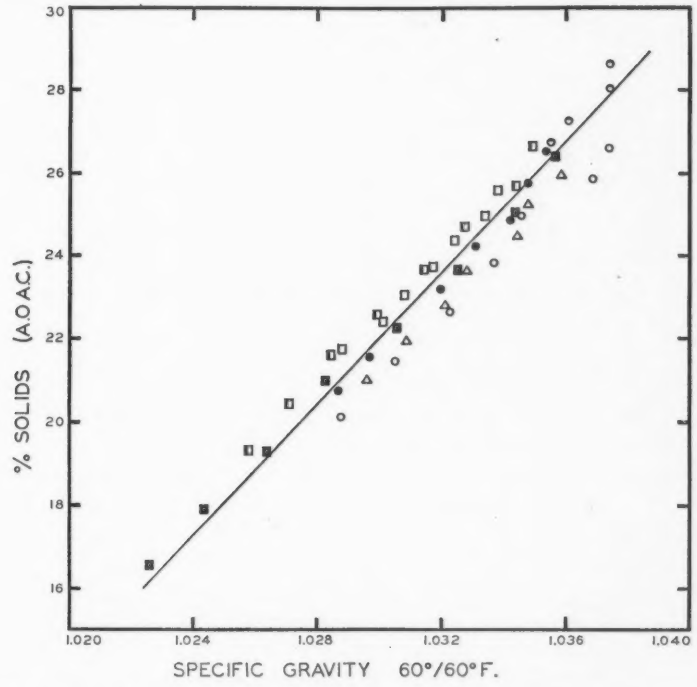

FIG. 1. *Relation between solids content of liquid egg and specific gravity.*

Key

- □ *Grade A large summer eggs obtained within one day of laying.*
- ■ *Currently available Grade A large summer eggs.*
- ◧ *Currently available Grade A pullet summer eggs.*
- ○ *Grade A large spring eggs held three months at 0° C. (32° F.).*
- ● *Grade A large spring eggs held three months in commercial storage.*
- ◓ *Grade A large spring eggs held three months at 0° C., then two weeks at 21.1° C. (70.0° F.).*
- △ *Grade C summer eggs.*

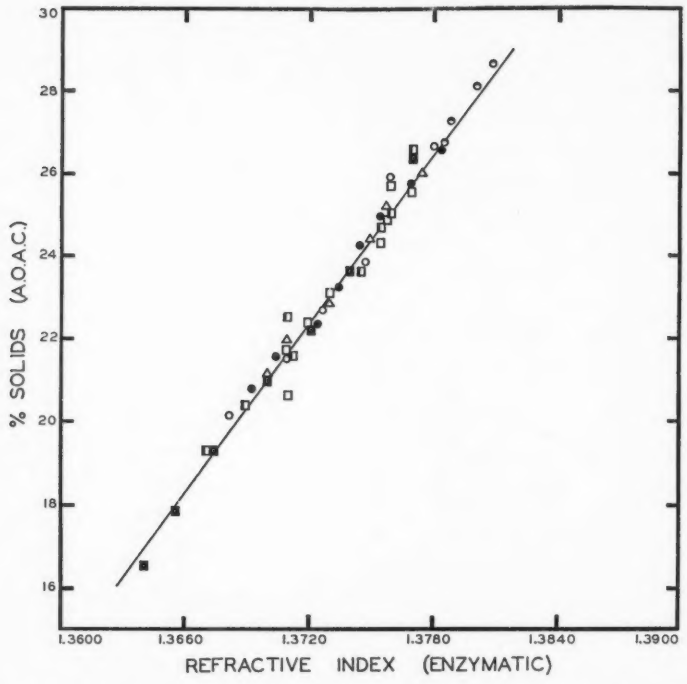

FIG. 2. *Relation between solids content of liquid egg and refractive index after treatment with trypsin (Key in caption for Fig. 1.).*

did not hinder combination of the data to establish the mean relations shown in the figures. These equations are based on equal numbers of determinations performed on identical samples, and are of value chiefly for comparative purposes. Best estimate equations recommended for predicting solids content from refractive index (electrolytic method) are discussed later.

The error in determining solids content by the specific gravity method was of sufficient magnitude to preclude its use, except as a rough check. It was evident that differences resulting from previous history exerted a pronounced influence on the relation obtained. This method was unsatisfactory for frozen egg owing to the lack of homogeneity in the thawed material. Attempts at homogenizing the melted egg resulted in the incorporation of air into the product, which did not pass off even on long standing, thereby preventing accurate determinations.

Both refractometric methods were relatively satisfactory (Table I and Figs. 2 and 3). Refractive index measurements on liquid egg after treatment with ammonium hydroxide was the most satisfactory of the methods investigated. The accuracy of prediction was somewhat better. The use of ammonium hydroxide rather than the trypsin solution was simpler from the

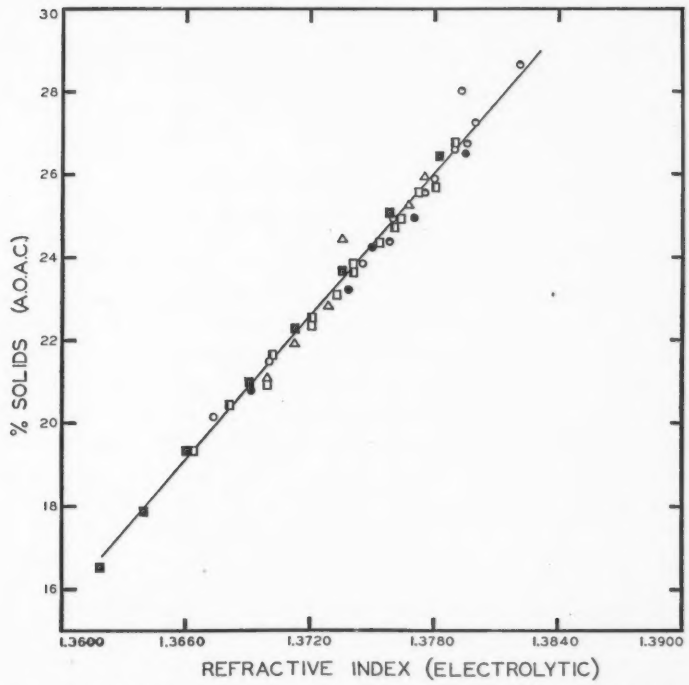

FIG. 3. *Relation between solids content of liquid egg and refractive index after treatment with ammonium hydroxide. (Key in caption for Fig. 1.)*

point of view of laboratory technique, and in addition gave a more easily readable field in the refractometer.

Before putting any of the above determinations into practical use as a standard method of measuring solids in liquid egg, it would be advisable to evaluate differences of technique existing between laboratories. The calibration curves shown in Figs. 2 and 3 are approximately 0.5 and 1% lower respectively than those previously recorded (2, 4). This disagreement may be attributable to the different methods of obtaining liquid egg of varying solids content.

Best Estimate Equations

Since the initial comparison showed the refractometric method involving the use of ammonium hydroxide to be the best of those considered, it was used in all subsequent work. Further measurements were made using this method to include determinations on eggs produced over the major portion of the Canadian laying season in computing the final prediction equation. The method was also used on thawed frozen egg. Equations, and errors of prediction, for both liquid and melted frozen egg are given in Table II. These relations are shown graphically in Fig. 4. It will be noted that the equations

TABLE II

EQUATIONS, WITH ERRORS OF PREDICTION, RELATING PER CENT SOLIDS IN EGG (y) TO REFRACTIVE INDEX, ELECTROLYTIC METHOD (x)

Kind of egg	Equation	Error of prediction, % solids
Liquid	$y = 593.53\,x - 791.68$	$\pm$ 0.76
Frozen	$y = 587.08\,x - 781.56$	$\pm$ 0.48

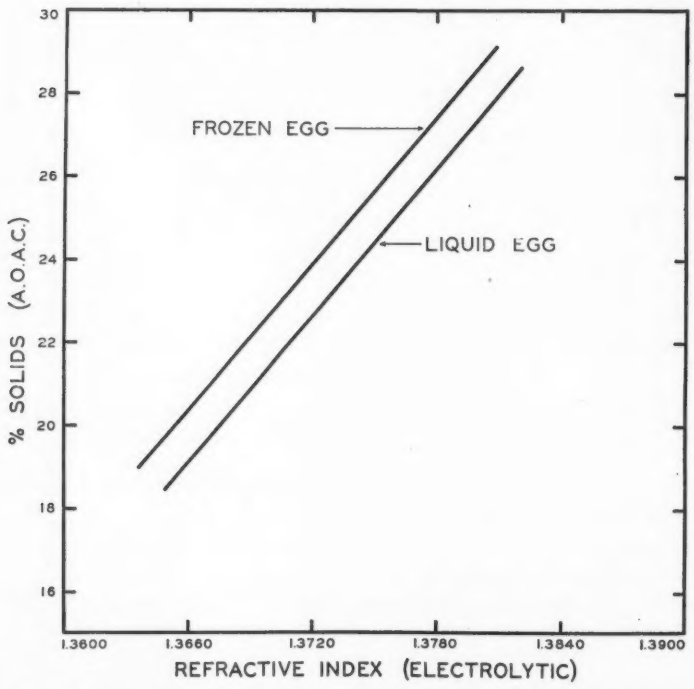

FIG. 4. *Relations between solids content of egg (liquid and frozen) and refractive index after treatment with ammonium hydroxide (as calculated from the best estimate equations).*

for liquid and frozen egg have approximately the same slope, but quite different intercepts, and so are not interchangeable.

This method of determining solids in egg is rapid, simple, and sufficiently accurate for industrial purposes.

Determinations Using a Hand Refractometer

The instrument used in the refractometric work so far described was not portable, and required a constant temperature bath. For routine inspection

a portable instrument would be very advantageous. For this reason, consideration was given to an easily obtainable, relatively inexpensive hand refractometer, designed to determine sugar concentration directly. No method of temperature control was provided for this hand instrument, but a correction thermometer reading in per cent solids for sugar was mounted on the side of the refractometer.

For a series of 18 readings at temperatures ranging from 19.0° to 32.0° C. (66.2° to 89.6° F.) the change of refractive index of liquid egg with temperature was observed to be −0.00012 refractive index units per °C. rise in temperature, (−0.00007 units per °F.). The corresponding figure for sugar solutions, as calculated from available data (3), is approximately −0.00014 (−0.00008). Since these two figures are nearly the same, the temperature correction given for sugar may be applied directly to egg.

Since the relation between refractive index and per cent sugar of sugar solutions is known (3), and that between refractive index (electrolytic method) and per cent egg solids is given in Table II, it was possible to calculate the equations given for converting readings on the hand sugar refractometer to per cent egg solids (Table III). Although these equations could be calculated it was necessary to know the error of prediction involved when using this hand instrument. To evaluate this, a series of 23 determinations was made on identical samples using both the hand and Zeiss sugar refractometers. This gave a direct comparison of the error involved in using each instrument. With this knowledge it was possible to calculate, by simple ratio, the error of prediction involved when using the hand instrument and the prediction equations given in Table III, because these equations were based on the same determinations as those given in Table II.

TABLE III

EQUATIONS, WITH ERRORS OF PREDICTION, RELATING PER CENT SOLIDS IN EGG (y) TO READINGS ON A HAND REFRACTOMETER (x)

Kind of egg	Equation	Error of prediction, % solids
Liquid	$y = 1.024\,x - 3.59$	± 0.56
Frozen	$y = 1.016\,x - 2.14$	± 0.35

The procedure for determining per cent solids with the hand refractometer would then be as follows: Prepare the sample and place on the prism as previously described; read the instrument (% sugar); read the temperature correction thermometer and apply the correction to the figure just read; convert the corrected figure to per cent egg solids using the equations given in Table III. This instrument, combined with the electrolytic treatment of the egg, provides a rapid, convenient, and inexpensive method for determining the solids content of egg in industrial practice.

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A SURVEY CAMERA TO FIT A BERGER TRANSIT[1]

By R. A. Nodwell[2] and R. C. Burstow[3]

Abstract

An accurate survey camera covering a horizontal angle greater than 45° on 3¼ by 4¼ in. plate or film is described. The camera is easily interchanged with the telescope of a Berger transit.

A small, accurate survey camera, which was designed and constructed at the request of the Department of Lands, Forest Branch, Province of British Columbia, is described in this paper. The camera takes 3¼ by 4¼ in. plate or film, covers a horizontal angle of approximately 50°, is interchangeable with the telescope of a Berger transit, and is light and compact for ease in transport.

Frequently lenses of very small *f*-numbers have been used in cameras of this type. Recent tests on lens–film resolving power of photographic lenses have indicated that best average photographic resolving power is obtained at medium apertures of *f*/11 or *f*/16. With further increase in aperture the aberrations become large and with decrease in aperture the loss in resolving power due to diffraction of light greatly exceeds the gain due to decrease in aberrations. This would indicate that the best aperture for the camera would be about *f*/11. However, a Ross 4 in. *f*/4 wide angle Xpres survey lens, which could be stopped down to *f*/16, was available, and, since a wider aperture might be useful at times in spite of decrease in resolving power, it was decided to equip the camera with this lens. The lens is mounted in a Compur shutter with a speed range from "Time" to "1/200 sec.".

Since any stray light that falls on the photographic plate reduces the contrast and resolving power, it is very important that adequate baffling be provided inside the camera body. A relatively large camera cone is required to accommodate this baffling. This makes it necessary to mount the camera with the lens located approximately between the uprights of the transit. Thus the centre of gravity of the camera is not over the axis of rotation, but it was found that the resulting stress did not impair the accuracy of the transit.

The accurate location of the photographic plate in the focal plane is accomplished in the following manner. The camera back, *A*, (Fig. 2), which retains the plate or film holder, is free to move about 1/16 in. in a direction parallel to the axis of the lens. The movement of this back is controlled by two cams, *B*, which are rotated by the handle, *C*. The camera back is pulled against the cams by two internal leaf springs. Thus with the back, *A*, in its forward position the plate or film is held in direct contact with the surfaces, *D*, of the

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camera body. These contact surfaces define the focal plane of the camera and hence the principal distance is independent of any dimension of the plate holder.

The principal point is defined by four fiducial marks, *E*. When the transit is level the principal axis is held horizontal by means of the screw, *F*, (Fig. 1), and an opposing screw that bears on a hardened steel bar mounted on the transit. The opposing screw is firmly held by a lock nut, which may be loosened in order to make small adjustments on the screw when the instrument is being checked in the field. The fiducial marks are so adjusted that the line joining the transverse marks is horizontal and that joining the other two is vertical.

The drum, *G*, which casts a silhouette of a number from 1 to 8 onto the plate or film, is rotated by an exterior wheel, *H*. This wheel displays a number corresponding to that being recorded on the film or plate.

The camera is equipped with three interchangeable filters. A ground glass in a suitable holder is also supplied.

Fig. 3 shows the camera and its accessories in the case.

Acknowledgment

The authors wish to express their appreciation to the members of the Optics and Metrology Sections who assisted in the construction and calibration of this camera.

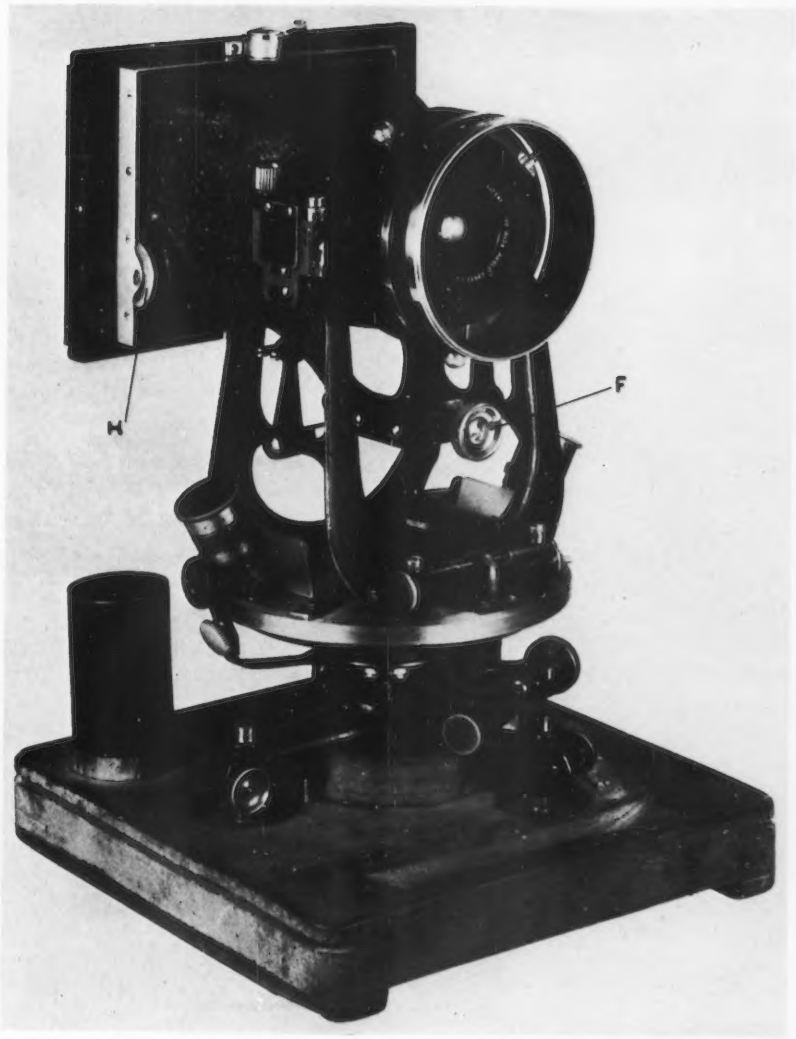

FIG. 1. *The camera mounted in the transit.*

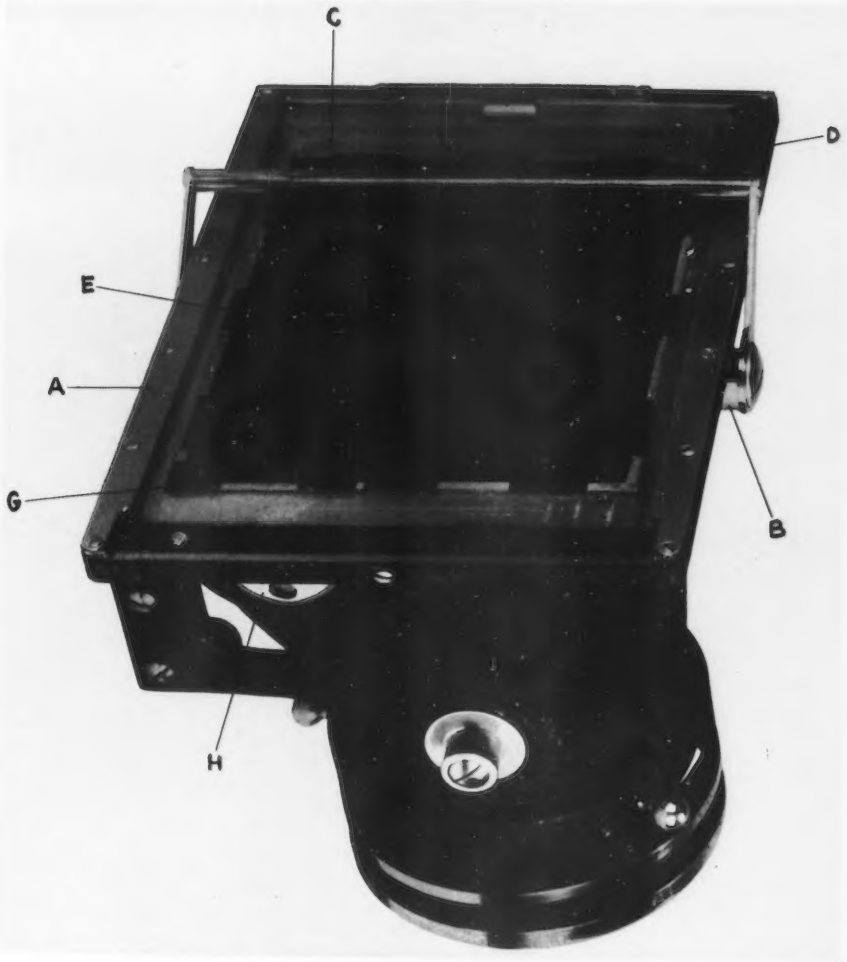

FIG. 2. *View showing back of camera.*

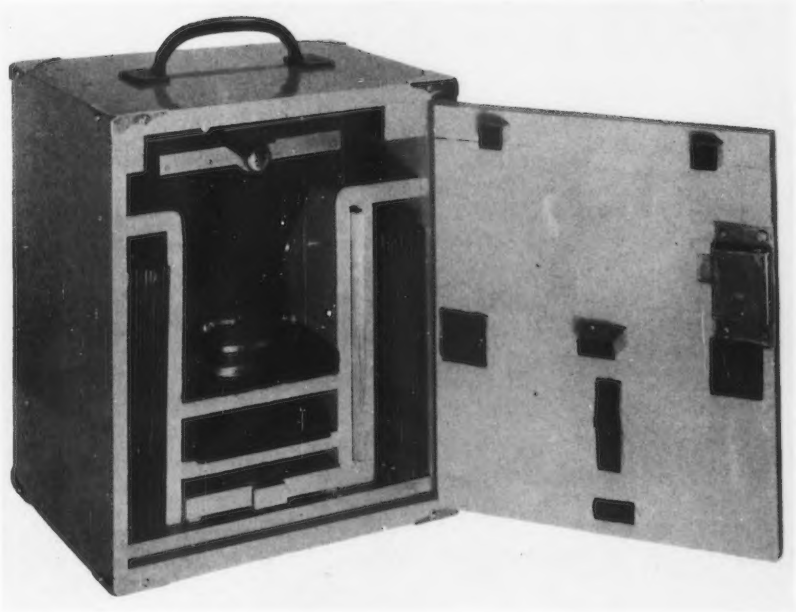

FIG. 3. *The camera and its accessories in the case.*

THE EFFECT OF WEATHERING ON COTTON FABRIC CONTAINING CERTAIN COPPER ROTPROOFERS[1]

C. H. BAYLEY[2] AND M. W. WEATHERBURN[2]

Abstract

On exposure to outdoor weathering for three months, No. 8 cotton duck showed substantial loss in breaking strength. The untreated fabric showed a loss of the same order as the losses of samples treated with copper naphthenate, copper hydroxynaphthenate, copper oleate, and copper tallate containing 0.1 to 1.0% copper. The copper treated samples showed slight evidence of increased actinic degradation as measured by cuprammonium fluidity. There was an appreciable decrease in the copper content of the treated samples on weathering. The decrease in copper content and breaking strength on weathering and the extent of attack by micro-organisms in soil burial testing were reduced considerably by the presence of a waterproofing compound of the wax-pigment-filler type. The initial water resistance of the proofing was modified by the presence of the copper compounds, being reduced by copper naphthenate, oleate, and tallate and increased by copper hydroxynaphthenate although on ageing and weathering these effects were minimized.

During the past four years considerable use has been made in the United States and Canada of organic compounds of copper as rotproofers for cotton fabrics. The more commonly used of these compounds include copper naphthenate, hydroxynaphthenate, oleate, and "tallate". Copper "tallate" is a mixture of copper derivatives of the organic acids present in "tall oil",* a product of the processing of southern pine (6).

It is well known that cellulose materials such as cotton duck used in tents, tarpaulins, etc., undergo actinic deterioration when exposed to sunlight for extended periods. It is also known that the presence of certain added constituents has an important effect on this actinic degradation. Whittaker (7) has studied the tendering effects of certain types of dyestuffs on cotton and rayon. On the other hand, chromium oxide precipitated on cotton cloth appears to reduce actinic degradation (4). Fargher has reported (5) that the danger of accelerated actinic degradation resulting from the presence of copper is small, and is more likely to occur in heavy, closely woven cloths than in thin open ones, probably owing to the slower rate of loss of copper from the former.

In view of the wide use of copper soaps as rotproofers it was of interest to determine the effect of such compounds on the actinic degradation of cotton fabric. The processors in Canada of tarpaulins for Service transport vehicles have utilized a waterproofing compound of the wax-pigment-filler type containing copper naphthenate applied to No. 8 cotton duck. Therefore, in this study use was made of copper naphthenate and other copper soaps applied

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* *The acids present in tall oil consist of fatty and resin acids, chiefly oleic, linoleic, linolenic, and abietic.*

to No. 8 cotton duck both with and without the addition of waterproofing materials.

Samples containing a range of copper contents were prepared and the effect of exposure to weather on breaking strength, cuprammonium fluidity, water resistance, and rot resistance determined.

Materials Used

The copper compounds used were commercial preparations of the naphthenate, hydroxynaphthenate, oleate, and tallate*.

The fabric used was unbleached No. 8 duck, all the samples being taken from the same length of fabric. The treatment of the samples was carried out on specimens 9 by 36 in. in the case of the laboratory samples, 180 by 36 in. in the case of the commercial samples. The samples containing the copper compounds alone were prepared in the laboratory as previously described (1). In the case of copper hydroxynaphthenate, which did not dissolve completely in Stoddard solvent, the treating mixture contained an appreciable amount of a pale blue precipitate. The samples carrying the pigmented proofing treatment in addition to the copper compounds were prepared by the Dominion Oilcloth and Linoleum Co. The colour of the latter samples, purple-brown, was obtained by the use of an iron oxide pigment. The proofing mixture containing the copper compound was applied in paste form by spreading. In proofings of this type the amount of material added to the fabric is approximately 40% on the weight of the original fabric, about one-half of this being wax. The proofing is one that completely seals the fabric, and the wax may be regarded as extending in a continuous film throughout the fabric.

The copper contents aimed at were 0.1, 0.3, 0.5, and 1.0%. These were readily obtained in the case of the laboratory samples but in the case of the commercial proofings the samples containing the higher percentages of copper showed somewhat lower amounts of copper than were desired. However, it was believed that the copper contents in the two series were sufficiently similar to permit a comparison of the results.

In this paper the term "treated" refers to the presence of various copper compounds alone, while the terms "proofed" or "unproofed" refer to the presence or absence of the waterproofing wax-pigment-filler mixture.

Test Methods

The weather exposures were carried out on the roof of the National Research Laboratories, Ottawa, from July 10 to October 10, 1944, the samples being attached to wooden racks and slung at an angle of 45°, facing southwest. Each exposure specimen consisted of duplicate samples sewn together with a double seam in the long dimension. The specimens were protected from

** While considerable use has been made in Australia of copper oleo-stearate as a rotproofer, this material has not been used to any appreciable extent on this continent, and was not investigated.*

contact with the wooden frames by means of 16 by 10 in. runners of unbleached duck sewn across the ends of the specimens, and were attached to the frames by cotton ropes fastened to grommets situated at the corners of the runners.

The methods of test used were those given in Schedule 4-GP-2-1944 of the Canadian Government Purchasing Standards Committee. Breaking strengths were determined by the 1 in. ravelled strip method, those on the controls being carried out on samples that had been leached in water to compensate for shrinkage resulting from wetting during weathering. Rot resistance was determined by the soil burial method, tests being carried out on: (*a*) samples that had been subjected to weathering, (*b*) samples that had not been weathered, (*c*) samples that had not been weathered but had been subjected to a period of leaching. The leaching treatment involved exposure of each sample (6 by 15 in.) in a bottle of 1 litre capacity to the action of water at 25° C. flowing at a rate of 10 litres per hour for 24 hr. The water resistance of the proofed samples was determined by the variable-head method.

Copper determinations were carried out by the ignition method (3). Determinations of cuprammonium fluidity were performed on samples from which the solvent-soluble portion of the copper compounds had been removed by extraction with Stoddard solvent, this measurement being made on the unproofed samples only since it was not possible to completely remove all the constituents of the waterproofing compound.

Data

(1) Weather Conditions

The data for weather conditions during the exposure period are given in Table I. It will be noted that the weather was fairly warm, with considerable sunshine, and not much rain, particularly in July and August. The conditions

TABLE I

RECORD OF WEATHER CONDITIONS DURING THE EXPOSURE PERIOD

Period	Mean daily temp., °F.		Rainfall, in.	Sunshine, total hr.
	Maximum	Minimum		
July 10 – July 16	82	59	0.53	56.6
July 17 – July 23	78	52	1.09	69.8
July 24 – July 30	81	62	0.74	45.8
July 31 – Aug. 6	88	63	0	77.6
Aug. 7 – Aug. 13	90	57	0	80.0
Aug. 14 – Aug. 20	85	59	0.25	62.9
Aug. 21 – Aug. 27	76	51	0.20	61.8
Aug. 28 – Sept. 3	77	56	0.60	45.4
Sept. 4 – Sept. 10	70	50	0.33	39.6
Sept. 11 – Sept. 17	78	55	0.74	32.6
Sept. 18 – Sept. 24	69	49	0.87	37.9
Sept. 25 – Oct. 1	55	43	1.80	32.3
Oct. 2 – Oct. 10	60	41	0.58	33.1
Average	76	54	Total 7.73	675.4

were, therefore, such as to favour actinic degradation. However, there was some evidence of superficial fungus growth on the underside of the untreated control sample of unbleached duck. The organism noted was a species of *Alternaria.*

(2) Breaking Strength Loss

The data are given in Table II, while Table III gives an analysis of variance of the breaking strength losses as given in Table II. It is apparent that the effects of proofing, treatments, and concentrations are highly significant. The following additional observations may be made from a study of the table, bearing in mind the differences specified as necessary (5% level of statistical significance).

(a) Weathering Effects

The samples containing copper hydroxynaphthenate showed lower breaking strength losses than did the samples containing the other compounds, this effect being more pronounced in the unproofed samples. On the whole, the losses shown by the samples containing oleate are somewhat greater than in the case of the naphthenate and tallate, particularly in concentration of 1% copper. In general the unproofed samples showed considerably greater losses than the proofed samples. The losses shown by the unproofed samples containing 0.1% copper are lower than those of the unproofed samples containing higher percentages of copper. This effect is not observed in the proofed samples, there being no indication that the losses are proportional to the copper content. Comparing the unproofed and proofed samples containing the various compounds with the untreated controls, it will be seen that there is no evidence that the presence of copper resulted in any marked increase in breaking strength losses, except possibly in the case of the sample containing 1.13% copper in the form of copper oleate (Sample OL 4).

(b) Burial Effects

When subjected to exposure followed by soil burial, the breaking strength losses shown by the proofed samples are considerably lower than those of the unproofed samples for all concentrations of copper and for all compounds investigated. In the case of the unproofed samples, the losses shown by those containing hydroxynaphthenate and tallate are similar, and are lower than those shown by the naphthenate and oleate, which are also similar.

When subjected to soil burial without previous exposure, the breaking strength losses of the unproofed samples containing copper naphthenate are considerably lower than those containing the other compounds, the latter being similar to each other. The proofed samples show losses similar to those of the unproofed, except in the case of the samples containing copper hydroxynaphthenate in concentrations of 0.1 and 0.5% copper, and copper oleate and tallate containing 0.1% copper, in which the losses of the proofed samples are lower. With regard to the effect of the copper content of the samples, the losses shown by samples containing 0.1% copper are considerably greater than those containing higher concentrations of the metal, the only exception

TABLE II

BREAKING STRENGTH LOSSES

Sample	Original copper concentration, %	Breaking strength loss, %, after			
		Exposure 3 months	Exposure and burial 2 weeks	Burial 2 weeks (unleached, unexposed)	Burial 2 weeks (leached, unexposed)
A. Not proofed					
Untreated					
UN	0	36.9	73.2	96.5	—
Copper naphthenate					
N1	0.096	18.7	36.9	0.8	45.2
N2	0.31	34.5	39.6	+10.4	44.2
N3	0.48	43.3	44.5	1.2	47.8
N4	0.98	37.7	43.2	0.8	18.9
Copper hydroxynaphthenate					
HN1	0.13	10.8	23.7	37.3	80.9
HN2	0.31	18.3	25.5	21.5	45.8
HN3	0.64	30.2	26.3	9.4	52.9
HN4	1.10	26.6	33.6	12.5	46.9
Copper oleate					
OL1	0.090	28.0	37.2	36.5	64.4
OL2	0.32	44.0	46.8	8.6	35.6
OL3	0.58	43.0	44.5	11.0	22.8
OL4	1.13	53.3	52.2	4.7	1.2
Copper tallate					
T1	0.072	27.7	22.1	37.8	49.4
T2	0.34	37.5	33.3	18.0	42.6
T3	0.54	46.4	39.2	3.1	38.7
T4	1.08	36.4	35.3	2.2	10.1
B. Proofed					
Untreated					
PUN	0	24.5	69.8	94.9	93.0
Copper naphthenate					
PN1	0.093	24.5	18.1	2.3	6.8
PN2	0.21	12.1	9.8	+ 4.5	+ 6.9
PN3	0.37	18.4	18.8	10.2	5.6
PN4	0.62	22.2	18.6	+11.7	+ 2.8
Copper hydroxynaphthenate					
PHN1	0.077	17.8	15.9	20.0	14.8
PHN2	0.17	13.5	15.8	3.1	+ 1.9
PHN3	0.32	13.1	13.8	+ 3.0	4.5
PHN4	0.74	18.6	9.9	+ 4.9	5.3
Copper oleate					
POL1	0.074	18.1	10.6	18.9	5.5
POL2	0.25	20.0	12.9	7.5	3.5
POL3	0.36	22.2	15.9	11.9	7.9
POL4	0.76	26.4	24.0	7.7	+ 8.7
Copper tallate					
PT1	0.088	13.5	10.0	26.0	19.5
PT2	0.22	20.6	17.6	17.6	11.2
PT3	0.41	27.9	17.9	13.0	1.1
PT4	0.83	17.8	11.2	+ 9.1	+ 6.6

TABLE III

ANALYSIS OF VARIANCE OF BREAKING STRENGTH LOSS

Source of variation	Degrees of freedom	Mean square			
		Exposure 3 months	Exposure and burial 2 weeks	Burial 2 weeks (unleached, unexposed)	Burial 2 weeks (leached, unexposed)
Proofed vs. not proofed	1	1596.13**	3678.68**	253.12*	10738.45**
Treatments	3	248.28**	170.93**	417.70**	328.68*
Concentration	3	187.97**	71.68**	698.22**	1052.53**
Treatment × concentration	9	27.41	24.96	99.90*	97.72
Proofed × treatment	3	75.92*	98.04**	111.85*	224.22
Proofed × concentration	3	119.58*	27.31	72.00	247.19*
Proofed × treatment × concentration	9	18.92	9.33	24.73	61.12

** *Significant at 1% level.*
* *Significant at 5% level.*

being copper naphthenate, which gave good protection even in 0.1% copper concentration.

In certain instances there was an apparent increase in strength after burial. This might be attributed to variation in the strength of the original fabric or to shrinkage brought about by burial which was greater than that produced by the leaching treatment of the control samples. In any case, these values are not statistically significant.

When subjected to leaching in water followed by soil burial the breaking strength losses shown by the unproofed naphthenate, oleate, and tallate treated samples are similar and lower than those shown by the samples containing the hydroxynaphthenate. The breaking strength losses of the unproofed samples containing 1% copper are lower than those for samples containing 0.3 and 0.5% copper, the latter being similar and lower than the losses of the 0.1% copper samples. With the proofed samples the losses are very much lower, particularly in cases where the losses of the unproofed were appreciable, namely, the hydroxynaphthenate in all concentrations, the others in concentrations below 1.0% copper.

(3) Loss of Copper

Measurements of copper losses resulting from the various test procedures were carried out on the samples originally containing approximately 0.3% of the metal (Table IV). It will be noted that exposure resulted in considerable loss of copper (63 to 81%) from the unproofed samples, and also that the losses were considerably less (5 to 24%) with the proofed samples. With the unproofed samples the losses of copper were increased slightly (73 to 93%) as a result of burial following exposure. With the latter samples the copper losses resulting from burial with preliminary leaching were considerably less

TABLE IV

LOSS OF COPPER

Sample	Original copper conc., %	Copper, %, on fabric after				Copper loss, %, after			
		Exposure 3 months	Exposure and burial 2 weeks	Burial 2 weeks	Leaching and burial 2 weeks	Exposure 3 months	Exposure and burial 2 weeks	Burial 2 weeks	Leaching and burial 2 weeks
N2	0.31	0.058	0.022	0.25	0.17	80.7	93.0	19.4	45.2
HN2	0.31	0.10	0.082	0.23	0.18	67.8	73.5	25.8	41.9
OL2	0.32	0.12	0.087	0.22	0.22	62.5	72.8	31.3	31.3
T2	0.34	0.11	0.082	0.28	0.21	67.7	75.9	17.7	38.2
PN3	0.37	0.28	0.28	0.28	0.26	24.3	24.3	24.3	29.8
PHN3	0.32	0.29	0.30	0.30	0.27	9.4	6.3	6.3	16.6
POL2	0.25	0.20	0.20	0.20	0.20	20.0	20.0	20.0	20.0
PT3	0.41	0.39	0.36	0.36	0.34	4.9	12.2	12.2	17.1

than those resulting from exposure alone. Burial without preliminary leaching gave similar losses for both the unproofed and proofed samples, except for the sample containing copper hydroxynaphthenate, but, with samples leached before burial, the presence of the wax proofing decreased the extent of copper loss resulting from burial.

(4) Water Resistance

The measurements of water resistance of the proofed samples were carried out a few days after the application of the proofing treatments, again at the end of the three month exposure and also on a set of unexposed samples that had been stored for three months in the laboratory. The data are given in Table V. It will be observed that the original water resistance values for the samples containing copper hydroxynaphthenate were higher than those for the untreated control, whereas the values for the other three compounds were lower, this being particularly noticeable in the samples containing more than 0.1% copper. In the case of copper oleate, there was a marked decrease in water resistance with increase in copper content. The water resistance of the samples containing naphthenate, oleate, and tallate increased as the result of weathering and, to a lesser degree, on storage, whereas that of the hydroxynaphthenate decreased. The water resistance of all samples after exposure, with exception of those containing 0.36 and 0.76% copper as copper oleate, were at least equal to that of the original untreated sample.

(5) Cuprammonium Fluidity

It will be noted from Table VI that the fluidity tends to increase with increase in copper concentration, except in the sample containing copper hydroxynaphthenate. While the rise in fluidity shown by the other three compounds roughly parallels the breaking strength loss, the increase in fluidity shown by the samples does not appear to be of the same magnitude as the breaking strength loss. This loss in strength cannot be attributed to microbiological attack during exposure. It had been thought that the loss may have

TABLE V

EFFECT OF WEATHERING AND STORAGE ON THE WATER RESISTANCE OF WAXED FABRICS

Sample	Treating compound	Copper, %	Waterproofness, cm. pressure to cause leakage			Change, cm.	
			Original	After storage 3 months	After exposure 3 months	After storage	After exposure 3 months
PUN	Nil	0.0	71	79	77	+ 8	+ 6
PN1	Copper naphthenate	0.093	67	70	85	+ 3	+18
PN2		0.21	54	62	71	+ 8	+17
PN3		0.37	62	70	73	+ 8	+11
PN4		0.62	53	69	85	+16	+32
PHN1	Copper hydroxy-naphthenate	0.077	92	93	75	+ 1	−17
PHN2		0.17	80	78	73	− 2	− 7
PHN3		0.32	91	80	71	− 9	−20
PHN4		0.74	92	76	76	−16	−16
POL1	Copper oleate	0.074	60	69	78	+ 9	+18
POL2		0.25	44	54	77	+10	+33
POL3		0.36	39	47	67	+ 8	+28
POL4		0.76	31	35	50	+ 4	+19
PT1	Copper tallate	0.088	80	82	77	+ 2	− 3
PT2		0.22	66	67	77	+ 1	+11
PT3		0.41	65	70	76	+ 5	+11
PT4		0.83	61	61	75	0	+14

been the result of flexing of the samples by the wind during exposure. However, more recent data have shown that similar losses are obtained with samples held taut during exposure. This effect is receiving further study.

Discussion of Data

It is of interest to refer to a few points arising out of the above data, and in this connection attention is drawn to an apparent anomaly in the breaking strength data in Table II. It will be noted that, except with the samples containing approximately 0.1% copper in the form of naphthenate and hydroxynaphthenate, soil burial following exposure did not result in any marked increase in breaking strength loss above that produced on exposure alone, although soil burial of the unleached original samples resulted in marked loss in strength in the hydroxynaphthenate, oleate, and tallate treated samples. It would thus appear that as a result of exposure the samples had become more resistant to microbiological attack. This effect is also shown by the untreated control which lost 36.9% of its strength as a result of exposure, 73.2% on exposure followed by soil burial, and 96.5% as a result of soil burial alone. The effect is also shown by the proofed samples. It is possible that this effect is connected with the presence in the fabric of non-cellulosic materials—e.g., waxes, pectic substances, traces of salts, sugars,

TABLE VI

EFFECT OF WEATHERING ON CUPRAMMONIUM FLUIDITY OF UNWAXED SAMPLES

Sample	Treating compound	Copper, %	Fluidity		Increase in fluidity
			Original	After exposure	
Untreated			3.1	8.6	5.5
N1	Copper naphthenate	0.096	4.9	9.5	4.6
N2		0.31	4.8	11.9	7.1
N3		0.48	4.1	13.9	9.8
N4		0.98	4.3	14.9	10.6
HN1	Copper hydroxynaphthenate	0.13	3.7	9.5	5.8
HN2		0.31	3.8	9.8	6.0
HN3		0.64	3.7	10.2	6.5
HN4		1.10	3.7	10.3	6.6
OL1	Copper oleate	0.090	3.8	11.7	7.9
OL2		0.32	4.8	14.3	9.5
OL3		0.58	4.6	14.4	9.8
OL4		1.13	4.6	14.7	10.1
T1	Copper tallate	0.072	4.1	10.5	6.4
T2		0.34	4.3	11.5	7.2
T3		0.54	4.1	12.9	8.8
T4		1.08	6.4*	14.4	8.0

** Difficult to completely remove copper tallate by solvent, possibly owing to oxidation of unsaturated constituents.*

NOTE: *Cuprammonium fluidity expressed as reciprocal poises.*

etc., which tend to promote the growth of cellulose-destroying organisms present in the soil. Reference to the effect of such non-cellulosic materials in promoting the growth of fungi on cotton fabrics has been made by Fargher (5), who stresses the desirability of complete scouring in the case of cotton fabrics to be used under conditions conducive to microbiological attack.

It will be seen that burial of the original samples which had been previously leached resulted in a loss of strength much greater than that of the unleached samples. A recently completed study of this effect (2) has shown that copper naphthenate treated fabrics that have been subjected to sustained contact with water—e.g., soaking in running water for 24 hr.—lose much of their resistance to attack by soil micro-organisms. This effect appears to be associated with the conversion of a portion of the copper compound into a solvent-insoluble form, probably as a result of hydrolysis. It is probable that this effect also occurs with other copper soaps.

The data for the water resistance of the proofed samples are of interest in view of the marked changes in water resistance resulting from weathering and storage.

It may be mentioned that the effect of copper naphthenate in reducing the water resistance of the proofed samples is in agreement with observations made by commercial proofers.

Attention is drawn to the beneficial effect of the proofing compound in reducing the loss in strength and loss of copper resulting from weathering, and also in increasing the resistance of the samples to microbiological attack.

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